STANDARDISATION OF FLEXURE TESTING OF ENGINEERING CERAMICS

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Abstract

With the increase in usage of engineering ceramics, a new industrial standard is required in order to evaluate its properties and to perform a fair and just trade. The thesis investigates the faults and omissions of existing work and judges today's requirements thereby constructing a framework with which today's and future standards in flexure testing can be based.

The draft standard presented in this thesis covers the three major testing methods for determining the biaxial flexural strength (modulus of rupture) of engineering ceramics. The ring-on-ring, ball-on-ring, and 4-Ball test fixtures were all adopted as standard, since it is known that each of these systems is suited for a particular application and each has different advantages and disadvantages.

The three major biaxial test methods prescribed in this draft standard have been devised so that more consistent and accurate test results can be obtained. However, the uncertainty of measurement in flexure testing always exists and needs to be estimated.

The estimation of uncertainty in flexure testing in this study is based on the methodology provided in the ISO Guide to the expression of uncertainty in measurement. The results of the estimation showed that the uncertainty in measurement for the biaxial flexure test standard proposed in this thesis is very low compared to the inherent variability of the strength of ceramic materials.

It was also found that the applied load, thickness of the disc plate, and random effects are the three major components contributing to the overall uncertainty. The total uncertainty of measurement in biaxial flexure testing can be significantly minimised by the reduction of the uncertainty contributed from these components, especially from random effects.

Notation

a	unit area
A	area
A_E	effective area
b	beam width
d	diameter or beam depth
E	Young's modulus
F_f	frequency of failure
I	moment of inertia
l	length
k	coverage factor
m	Weibull modulus
M	bending moment
N	number of specimens
p	pressure
P_f	cumulative probability of failure
P_S	probability of survival
R_L	radius of the load ring
R_O	radius of the disc plate
R_S	radius of the support ring
R'_L	contact radius of the ball
s	standard deviation
t	thickness
T	Temperature
$u(x_i)$	standard uncertainty
$u_c(y)$	combined standard uncertainty
U	expanded uncertainty
v	unit volume
V	volume
V_E	effective volume

V_{eff}	effective degrees of freedom
W	applied load
W_f	fracture load
x_i	input estimate
X_i	input quantity
y	an estimate of the measurand Y
α	coefficient of linear expansion
$\dot{\varepsilon}^{o}$	loading rate
ρ	density
σ	stress
σ_f	fracture stress
σ_{max}	maximum stress
σ_n	principal stress
σ_{nom}	nominal stress
σ_o	normalising factor
σ_u	threshold stress
$\overline{\sigma}_f$	mean fracture stress
$\overline{\sigma}_{fa}$	mean failure stress of a specimen of unit surface area
$\overline{\sigma}_{fv}$	mean failure stress of a specimen of unit volume
$\overline{\sigma}_{nom}$	mean nominal failure stress
$\overline{\overline{\sigma}}$	median fracture stress
Δ	standard error
μ	coefficient of friction
ν	Poisson's ratio
ν_b	Poisson's ratio of the ball
ω	deflection
$\Sigma(A)$	stress-area integral
$\Sigma(V)$	stress-volume integral

CHAPTER 1
Introduction

1.1 Background

Engineering ceramics are potentially attractive as structural materials because of their high strength-to-weight ratio, high stiffness, high corrosion resistance, excellent high temperature properties, low friction, and abundant availability. The development and application of engineering ceramic materials has been the focus of much attention recently.

However, ceramic materials are not only characteristically brittle, but they as yet do not possess the high standards of uniformity, reproducibility, and reliability that are required of conventional structural materials. One of the major obstacles to the extended use of ceramics is the lack of design data. Part of this problem can be traced to the lack of standardisation of test methods for ceramics.

An accurate and standardised test method for the measurement of strength of engineering ceramics is desirable for several reasons. First, it is important as a technique for quality control in products and as a means for determining whether ceramic materials meet the strength specifications required by the user. Second, it is important in the development of ceramic technology to evaluate the effect of new raw materials or of variations in processing undertaken for the purpose of reducing costs or improving properties. Third, it is important for studies aimed at determining the factors such as processing, machining, microstructure, surface condition and environment, which affect the strength.

The Collyear [1] Report in the United Kingdom stressed, some years ago, the importance of test methods standardisation. The development of standard test methods has become one of three main lines of standardisation activities in the United Kingdom.

As a consequence of the cost and difficulty of conducting direct tensile testing on engineering ceramics, the strength of engineering ceramics is often

measured by the well-known flexure test method. Porcelain manufacturers came to use the test in the 1920s but insufficient. In the 1950s and 1960s flexure testing became a common tool of ceramic manufacturers and research laboratories. Flexure testing was, and still is, a low-cost, simple, and versatile method to assess the strength and quality of ceramic materials [2].

Several test techniques for flexure testing of engineering ceramics have been developed. There are many similarities among the techniques. Nevertheless, a myriad of test configurations arose with various specimen sizes and shapes, fixture sizes and types. There was little consistency in procedures or results. In order to obtain more consistent and accurate test results, the standardisation of flexure testing of engineering ceramics must be carried out.

1.2 Objectives

The objectives of this study are several fold: to understand the characteristic of engineering ceramics; to assemble the reported data on the flexural strength in literature; to compare the difference between the flexure testing methods; to establish a standard flexure test method which can be easily performed and possesses accurate and consistent flexure testing results; to measure the flexural strength of some commercial ceramic samples; to set up standardised data bases on the flexural strength of engineering ceramics; to develop a methodology with which a draft standard can be formulated; to draft the testing method standards, and then offer the results to the British Standards Institution, the European Committee for the Standardisation and the International Organization for Standardisation to establish BS, EN and ISO standards.

To achieve these objectives, firstly, the standardisation activities and the characteristics of engineering ceramics were reviewed. Secondly, the existing flexure test methods and the factors influencing the strength were studied. Thirdly, a series of step-by-step experiments were carried out in which the effects of testing parameters on the accuracy of flexure testing were investigated. Finally, based on these results, the important characteristic features governing flexure testing were determined and appropriate flexure test standards were

proposed.

1.3 Format

The work consists of several distinct aspects of standardisation of flexure testing of engineering ceramics. This was taken into account when writing this thesis by devoting a separate chapter to each major point. To maintain continuity, a general description to each aspect was included at the beginning of each chapter with a summary at the end.

The ultimate objective of the work is to develop a methodology whereby the flexure test method standards can be formulated in order to improve the accuracy and consistency of flexure testing results of engineering ceramics. Therefore, chapter one is devoted to the standardisation of engineering ceramics in which the development of engineering ceramics, the essential features of standardisation, the standard need in engineering ceramics, and the present situation of standardisation are reviewed.

Chapter two provides an overview of the measurement of flexural strength of engineering ceramics. Known measuring methods for flexural strength are briefly described. The statistical treatment of test data is outlined. The factors influencing the strength are discussed in detail.

Chapter three reviews the current uniaxial flexure test techniques, i.e. beam bending tests. The standardisation situation of beam bending tests is described. The comparison of existing standards is outlined. The errors associated with beam test are also discussed.

In the following chapters, the theoretical analysis and experimental investigation of three major techniques of the biaxial flexure tests for ceramics are described. The development of standard testing methods is also described. The estimation of uncertainty of measurement in flexure testing is discussed.

The final chapter in the thesis summarizes the work and lists the most important conclusions. Suggestions of areas for future work are given.

The structure of the thesis is outlined in Fig. 1.1.

Where possible the mathematical proofs and derivations are taken into Appendices at the end of the thesis to avoid disturbing the flow of the text. Experimental results and tables of numerical values are also given at the end for the same reasons.

Text pages follow consecutively throughout the thesis starting from the first chapter. Figures and tables are numbered consecutively within each chapter but are prefixed by the chapter number; both are included immediately after their first mention in the text.

Reference is made to published literature by bracketing a reference number after the appropriate section of text. The source of the information is given at the end of the thesis.

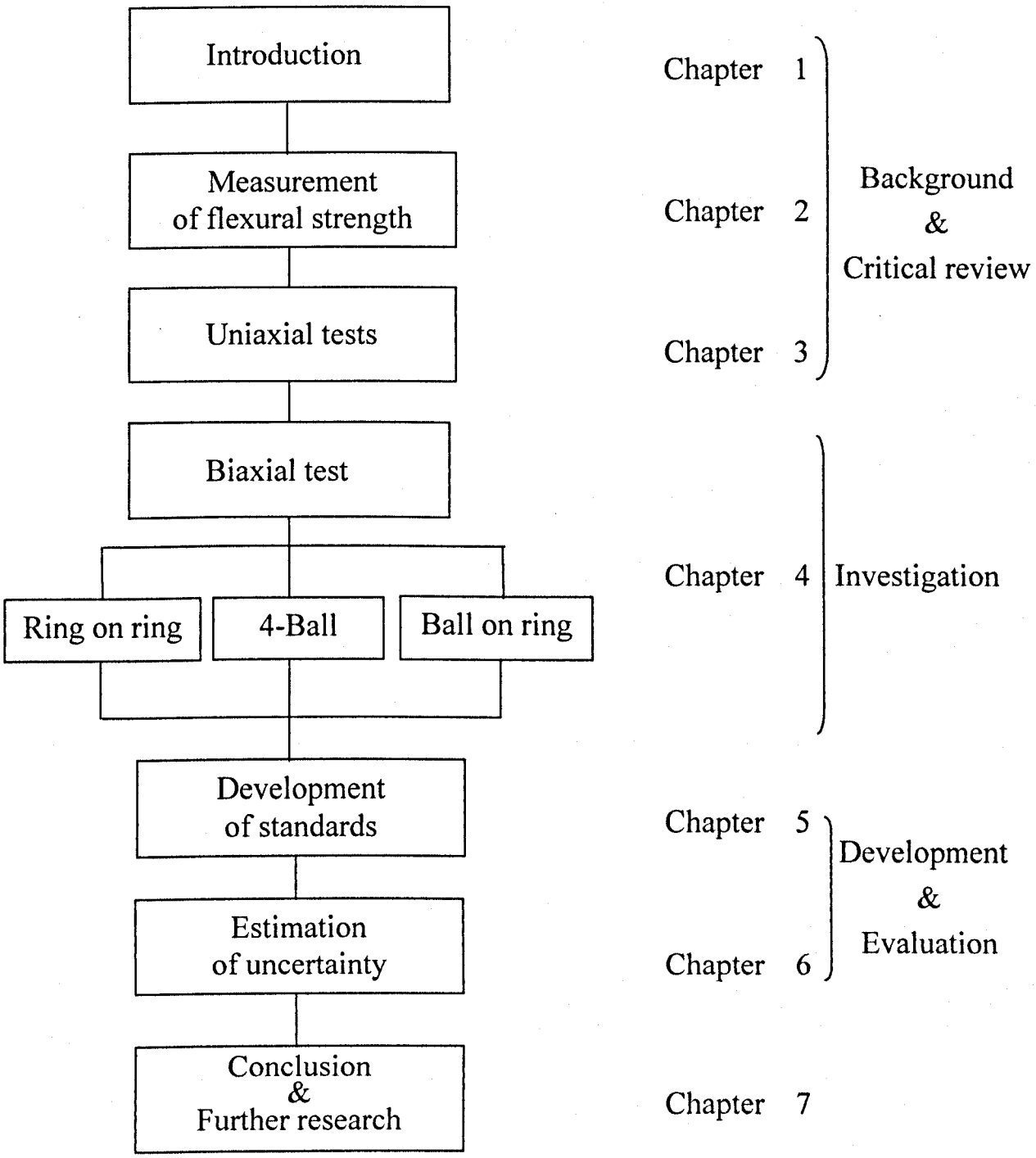


Fig. 1.1 Schematic structure of the thesis

1.4 Standardisation of engineering ceramics

Standardisation is an activity giving solutions for repetitive application, to problems essentially in the sphere of science, technology and economics, aimed at the achievement of the optimum degree of order in a given context. Generally, the activity consists of the process of formulating, issuing and implementing standards [3].

Standardisation is not a new expression which may be interpreted literally since nature itself has shown the path for the discipline of standardisation. For example, nothing is more perfectly standardised than the atom of oxygen or the molecule of water; also, on a higher level, the suns and their planets, or proteins which make living material; finally, being themselves. Sanders [4] states that nature, as it assembles particles, fits celestial bodies into space, populates the earth with human beings, and carries everything out according to pre-determined rules.

What is new about standardisation is the twentieth-century approach to the subject. In an ever enlarging civilized world, demanding better communications, more and more trade between nations and an insatiable demand for manufactured goods and appliances, standardisation has emerged both as a key to open many doors and also as a discipline which must be accepted by any civilized community if it is to enjoy the goods and services which it is now demanding. This has led us in the twentieth century to a whole new science of standardisation and to the development of product standards, firstly at a national level and later at an international level.

Standardisation is now an essential feature of economic development; a process and a tool. As a process it means the formulation of technical agreements required for communication between levels of activity or along chains of responsibility. As a tool it means the formulations themselves - dimensional agreements for example, recognized test procedures, specified material and performance requirements, specialized technical language and other rationalizing elements in the professional world [5].

One conviction shared unanimously by industry and the relevant government authorities concerns the utility of standardisation and the need to

strengthen it, since it provides so many advantages. By establishing a frame of reference for assessing advanced materials, it very largely eliminates the uncertainties concerning products and stabilizes the production process. Accordingly, it contributes to the promotion of domestic business and to the growth of international trade. As it speeds up the diffusion of products, it is a factor of growth in a context of harmony and safety (by protecting the consumer and the environment, guaranteeing the quality of products and their non-toxicity, etc.).

More common and objective methods for evaluation of materials will be possible by establishing quality standards as well as by promoting standardisation of evaluation methods. It will also provide a technical basis on which manufacturers and users could improve their interaction and collaboration. Standardisation has a great significance on development of engineering ceramics applications.

In the following, the features of ceramic material important to the engineer are highlighted. The basic concepts of standardisation are also reviewed, such as the aims and principles of standardisation, the structure of standardisation space, and the procedures for formulating a document standard. In addition, the standard need in engineering ceramics is described. Finally, the present situation of standardisation in Japan, the United States, Europe and on an international level are presented.

1.4.1 Development of engineering ceramics

Broadly speaking, "engineering ceramics" are ceramic materials used for engineering purposes. They typically are highly engineered, high performance, predominantly non-metallic, inorganic material that have specific functional attributes.

As compared to traditional ceramics that are made with firing of natural inorganic material such as china and porcelain, glasses and cement, etc., engineering ceramics are made with artificial materials such as alumina, zirconia and silicon nitride. Therefore, engineering ceramics are possible to have excellent thermal, mechanical and chemical properties, according to control material, chemical composition and process.

1.4.1.1 History of the engineering ceramic industry

The continuing demand for improved performance from engineering components has led to a reassessment of some materials that were previously considered unsuitable for engineering purposes. Ceramics are one such group of materials. Over recent years, the interest in ceramic materials has grown rapidly, particularly for applications involving high temperature or corrosive environments. In response to the demand, a number of new fine ceramics have been produced specifically for engineering purposes.

It was said (Kanno, 1996) that the research and development of fine ceramics that exploit the material's electric and magnetic features were stepped up in around 1930, when its electromagnetic features were first learned. Uses of these functional ceramics have grown since the 1950s when they became an ingredient in spark plugs, capacitors, integrated circuit packages, various sensors, and others, along with the development of the electronics, machinery, information and telecommunications industries [6].

The development of fine ceramics for structural use, which contains features as heat resistance, extreme hardness, abrasion and corrosion resistance, is said to have originated during the Cold War period after World War II when the United States felt a sense of crisis with respect to the acquisition of rare materials, thus developing a new heat resistant material called "Thermit" [7].

Full scale research and development with respect to such structural-use engineering ceramics began in 1972 during the oil crisis when expectations were high regarding the improvement of heat efficiency due to heat resistance and the results of energy conservation. Full scale development started on heat and abrasion resistant structural materials beginning with the utilization of gas turbines followed by automobile parts and bearings.

In 1982, a new type of ceramics, namely non-oxide ceramics, pursued the fame of the conventional oxide ceramics. The academic circle publicized many reports commending this new type of ceramic for its superior characteristics. Non-oxide ceramics were thus winning applause as a key material for sophisticated industry and advanced technology and as an indication of the coming of the second "stone age" [8].

Ceramics materials have been put to practical use in various industry and

consumer products. Demand for fine ceramics has shown steadily growth year by year. According to the reports in the "Annual Giants in Ceramics" by the Ceramic Industry, advanced ceramics are on a growth trend, reaching USD 20.2 billion in 1994. The sales for the category of engineering ceramics which includes structural components such as wear parts, cutting tools and heat engine components were 32% in 1994 [6].

In its report "Technical Ceramics: World Market and Technology Survey", World Business Publications Ltd. of London, UK, predicted that the global market for technical ceramics materials is tipped to increase steadily to USD 20.9 billion by the year 2000 from its estimated 1995 total of USD 7.1 billion. The report also said that although the commercialization of the more sophisticated applications for technical ceramics has taken much longer than that originally estimated, the advantage to be gained from their use would ensure the industry continues to develop [9].

J. Aoi in 1996 stated that handling the new material, the fine ceramics industry has more issues to resolve than do other industries handling conventional materials that occupy an established position in industry as a manufacturing material. Such issues include improving characteristics and reliability of guaranteed quality and price reduction. Regardless of these hindrances, however, fine ceramics will continue drawing attention as an inherently superior material in terms of energy saving, environmental conservation, and resource saving; these are the important social issues to be addressed continually into the future.

For the future outlook of the engineering ceramics industry, the superior characteristics of engineering ceramics and its potential of adding various features allow us to consider that this new type of material deserves the name as one of the new products of the next generation. It is thus expected that engineering ceramics broadens the scope of its application, and gradually, will spread into our daily lives. In order to better achieve estimates of uncertainty it is necessary to review how the ceramics are produced.

1.4.1.2 Manufacture of engineering ceramics

The manufacture of engineering ceramics often starts from the powder

processing. The nature of the raw material has a major effect on the final properties of a ceramic component. Purity, particle size distribution, reactivity, polymorphic form, availability and cost must all be considered and carefully controlled.

Properly sized and pre-consolidated powders are now ready for forming into the required shapes. Different forming processes are used to shape the finished product by utilizing properties of raw materials and by controlling each step within the overall process. The major techniques for consolidation of powders and producing shapes are [10]:

1. Pressing: the process is accomplished by placing the powder into a die and applying pressure to achieve compaction. It includes uniaxial pressing (dry pressing and wet pressing), isostatic pressing (hydrostatic pressing or molding), hot pressing and hot isostatic pressing.
2. Casting: more frequently, the process is done by a room temperature operation in which ceramic particles suspended in a liquid are cast into a porous mold which removes the liquid and leaves a particulate compact in the mold. It includes slip casting, thixotropic casting and soluble-mold casting.
3. Plastic forming: the process involves producing a shape from a mixture of powder and additives that is deformable under pressure. Heat normally must be supplied simultaneously to pressure. It includes extrusion, injection molding, transfer molding and compression molding.
4. Other forming processes: some applications such as plate-fin heat exchangers require thin strips or structures of ceramics. Tape forming has been developed as an effective means of meeting these needs. The doctor-blade process is well established for fabrication of electronic ceramics for capacitors; it consists of casting a slurry onto a moving carrier surface and spreading the slurry to a controlled thickness with the knife edge of a blade. The slurry is then carefully dried, resulting in a thin, flexible tape that can be cut or stamped to the desired configuration prior to firing. Another important established approach is green machining. It refers to machining of a ceramic part prior to final densification while the material consists of compacted, loosely bonded powder.

The shapes resulting from the forming processes described above consist essentially of powder compacts that must be identified by high temperature processing before they will have adequate strength and other properties. Some processes combine forming operation and densification in a single step. These include hot pressing, chemical vapor deposition, liquid particle spray, and cementitious bonding.

Some ceramic parts can be fabricated to nearly net-shape by a proper method. However, more frequently, machining of some of the surfaces is required to meet dimensional tolerances, achieve improved surface finish, or remove surface flaws. Ceramic material can be removed by mechanical, thermal, or chemical action. Mechanical approaches are used most commonly. They can be divided into three categories: mounted abrasive, free abrasive, and impact.

Post-machining procedures have been developed to obtain further improvement in the properties. These include the following: annealing, oxidation, chemical etching, surface compression and flame polishing [10].

Details of the above processes for the manufacture of the engineering ceramics can be found in the literature and have been well documented and reviewed on several occasions [10,11]. Hence, they will not be dealt with in any greater detail here.

1.4.1.3 Characteristics and applications of engineering ceramics

Ceramic materials are becoming increasingly important in engineering uses particularly in applications where strength at high temperatures is required. The characteristics and applications of engineering ceramics have been well documented and reviewed on several occasions, e.g. Refs 11, 12, 13, 14 and are only briefly summarized here.

Generally, ceramic materials maintain their strength and chemical stability above 1000°C, far in excess of the range of most metals. Even at these temperatures the materials are still relatively unreactive and can withstand corrosive environments without undue deterioration.

The hardness of most ceramics is typically high; a particular form of boron nitride is the hardest man-made substance with a hardness and heat resistance

greater than that of the diamond. In many materials, this hardness acts together with wear resistance to create an extremely durable material. However, the great hardness can be disadvantage in the manufacturing and fabrication; where finished dimensions cannot be achieved before the firing stage, expensive diamond grinding is often necessary.

Low thermal expansions are also a feature of ceramic materials. This property is useful to the designer concerned with hot clearances but, of much more importance, it limits the level of thermal stress set up in the material. Therefore, not only does the material maintain its strength at high temperatures, but it can also withstand extreme thermal gradients without damage.

The primary disadvantage of ceramics is their brittleness i.e. their lack of toughness. Fracture is rapid, comes without warning and is usually catastrophic. This has limited the use of engineering ceramics in spite of the many other attractive qualities they possess. Some attempts are being made to improve the impact resistance of ceramics by introducing fibres into the matrix.

Many applications of engineering ceramics have been realized based on their high hardness, wear and corrosion resistance properties [15]. Alumina pairs are widely used as sealing disks in hot and cold water taps, where low friction has been achieved by specification of surface roughness from 0.6 μm to nanometric levels. Sand-blast nozzles of B_4C or SiC, sealing rings for pumps and equipment for transporting corrosive or abrasive liquids are the subject of mass production. SiC seems to be the favoured material for the pump and pipe liners.

A field of prospective growth is that of roller and ball bearing and shaft protection sleeve production. Fully dense silicon nitride particularly can obviously improve the performance of antifriction bearings. Ceramic bearings provide the possibility of operation at high temperatures ($\geqq$800°C) and under severe environmental conditions. A relatively new application is in parts for big valves like cones and ball plugs. Products of general interest are knives and scissors manufactured from transformation-toughened zirconia.

Important products of heat-resistant structural parts are kiln furniture from SiC, a product which requires much less space than conventional kiln furniture and other furnace parts. SiC burners including heat exchangers and nozzles

for gas and oil burners may develop into a substantial market. A commercial product is TiB_2 evaporators and rings of hexagonal BN for horizontal casting. The welding and steel hardening industry perhaps represent a future potential market for application of Si_3N_4 materials because of their good thermal shock resistance. In industrial heat exchangers, ceramic materials permit higher operating temperatures (1300~1350°C) than metallic alloys, resulting in energy saving during heat recovery.

Ceramic metal-working tool materials and coatings compete with metallic and cemented carbide tools. Al_2O_3 and Al_2O_3-TiC materials have been used for special applications for about four decades. Si_3N_4 and Sialons have been successful since the 1970s in turning and milling of cast-iron parts and Ni-based superalloys. Si_3N_4 tool tips can be used for high-speed interrupted cutting operations due to their impressive fracture toughness and impact resistivity under these conditions. The excellent properties of engineering ceramic tools do not yet correspond at present to their introduction in practice, although for special machining operations in the car and aircraft and industry, ceramics are well established.

The strong efforts to introduce ceramic parts in spark-ignition, as well as, in diesel engines during the last 10 years are aimed at increasing the performance of these engine types. A considerable introduction of ceramics parts has been achieved at present in addition to the combustion area. Cordierite honeycomb catalyst carriers are now widely used in cars with gasoline-fuelled spark-ignition engines. Ceramic catalyst carriers are also very important in the chemical industries. The use of hotter exhaust gases is realized by the ceramic portliner, consisting of porous Al_2TiO_5. Strong efforts are under way to introduce ceramic monolithic turbochargers for passenger cars, trucks, and armoured military vehicles as well.

The swirl chamber from Si_3N_4 or Sialon was introduced in diesel cars, e.g. by Isuzu, Toyota and Mazda. The rocker arm insert is a typical ceramic wear part, which has been introduced as a Si_3N_4 tip in Mitsubishi cars. Sealing rings of SiC, a mass product in conventional pumps, are now used in cooling water pumps due to their superior friction behavior, combined with less noise generation.

The ceramic gas turbine, especially the all-ceramic engine is undoubtedly the most challenging project in utilizing engineering ceramics for industry. The expected benefits are the high turbine inlet temperatures, up to 1350°C, the multi-fuel capability and the potential of low environmental pollution.

The increasing seriousness of global environmental problems also result in the use of engineering ceramic materials. Recently, as a result of the rapid expansion of international economic and social activities, environmental problems such as global warming and destruction of the ozone layer are beginning to appear. To solve these problems, an investigation of all systems and materials is necessary. In the area of materials, developing new engineering ceramics that can improve heat efficiency, increase energy savings and raise the potential for recycling is greatly needed.

1.4.1.4 Obstacles to commercialization of engineering ceramics

The market growth of engineering ceramics seems to be much more sluggish than was expected over the past 10 years. The Ceramics Committee of SAMPE Japan Chapter in 1990 discussed the actual status of such materials in Japan and the barriers to their commercialization. They pointed out three major problems–Economics, Reliability and Applicability–to be tackled for more efficient development of the market [16].

It is a fact that there are many instances where even the cost performance is higher when compared to materials now in use (mainly metals). Although it seems no easy task to specifically itemize the factors affecting economics in a generalized way, the various points listed below cover the relatively common issues:

1. The economics of scale are insufficient.
2. Raw materials and additives are costly.
3. The manufacturing processes are limited in comparison to those for plastics and metals.
4. Machining costs are high.
5. Inspection costs are at a level that cannot be ignored.

Another obstacle to commercialization of engineering ceramics is the lack of reliability in such material properties related to failure or fracturing during

service. Typically, these are strength and toughness. In addition, the possibility that a fracture mode could develop into a catastrophic failure makes the issue all the more significant. Toughness measurement methods for ceramics still remain unsolved. It is therefore quite difficult to solve the questions of reliability only by employing statistical analysis to evaluate product quality and characteristics.

One of the reasons that applicability or application technology was considered as an obstacle to commercialization is the fact that we are still uncertain about ceramics applications. Also, counted as other reasons are our limited experience with this material, the absence of concrete steps to put ceramics into practical use, and the fact that methodology has not been established yet, all of which may be traced to the immaturity of the market.

To solve the question based upon the aforementioned technical problems, the various issues listed below were proposed by the SAMPE Japan's Ceramics Committee [16].

1. Diversification of raw material qualities and grades, plus price cutting.
2. Production methods with higher economic efficiency and controllability.
3. Cost reduction in machining and joining.
4. Nondestructive inspection (NDI) and other product inspection
5. Quality standards and standardisation of evaluation methods.

To succeed in developing engineering ceramics into practical products, there are some problems that need to be researched at the level of basic science or technology. Considering that engineering ceramics are pretty much still in their infancy, these problems related to the toughening mechanism, interrelationships between processing conditions and product properties, and in-service behavior of ceramics should be solved by elucidating them through basic science and technology. These are essential issues in respect to the true nature of engineering ceramics. We must be patient about their gradual development over a possible protracted period of time. However, windfall progress from some major breakthroughs may also be expected. In particular, unearthing just one innovative guiding principle would greatly affect the viability of promoting commercialization.

To strengthen promotion of commercialization, there are various aspects of subjects that also need to be addressed regarding the social and business environment. The following are the major items pointed out by the SAMPE Japan's Ceramic Committee [16].

1. Strengthen the linkage and cooperation between manufacturers and users.
2. Strengthen the systems unifying standards and terminology as well as promoting standardisation.
3. Build a database including failure examples and develop a system
4. Maintain the interdisciplinary and inter-industrial cooperation system centering on linkage of industrial, governmental and academic fields.
5. Strengthen an international exchange system to develop the diversified and growing market.

Technological progress of engineering ceramics over the past 10 years has been quite large. However, the nature of that progress has been concentrated on manufacturing techniques. The portion of progress dedicated to engineering does not necessarily contribute to commercialization. In the outlook for the future, at last earnest steps toward commercialization of engineering ceramics are coming closer, certainly accelerated market growth can not be far away.

1.4.2 Essential features of standardisation

For many years, standardisation was widely regarded as of secondary importance; a desirable enough activity, provided that one could afford it. In many companies a standard department would develop in the good years and retract or be disbanded altogether during the lean years in the quest for economy. In fact, the opposite approach would have been more appropriate. Likewise, standards people at both the national and international levels became accustomed to the fact that their activities were generally regarded as desirable rather than vital. Too few people recognized that standardising was the most effective means of all of making real economies.

Today, the attitude towards standardisation has changed entirely. In line with rapid technological development, improved transportation methods and increased integration between companies, countries and even continents, the

desire to standardise has been replaced by a need to standardise.

During the past quarter of a century, the development of the field of standardisation as an independent discipline has been proceeding quickly. However, the contents of this discipline are not fully familiar to the majority of measurement scientists and technologists. It is an aim of this study to bridge the gaps between disciplines of standardisation and measurement through the application of flexure testing of engineering ceramics.

1.4.2.1 Aims and principles of standardisation

The principal aims of standardisation were defined some years ago by the ISO committee for the Study of the Principles of Standardisation (code name ISO/STACO) as the promotion of [4]:

1. Overall economy in terms of human effort, materials, power etc. in the production and exchange of goods.
2. The protection of consumer interest through adequate and consistent quality of goods and services.
3. Safety, health and protection of life.
4. Provision of a means of expression and of communication amongst all interested parties.

The definition of the aims defined by the ISO/STACO was fairly widely accepted at the time and is still valid, but it now calls for examination and considerable amplification in view of the mass development of standardisation at all levels.

By the British Standard Guide of BS 0: Part 1:1981, a standard for standards – general principles of standardisation, the present-day aims of standardisation can be summarized as that which provides technical criteria accepted by consensus, and that standards promote consistent quality and economic production. They rationalize processes and methods of operation, making communication and the exchange of goods and services easier. Their use gives confidence to manufacturers and consumers alike.

Standardisation involves both the preparation and use of standards. The main principles for this may be grouped under the following headings [3]:

1. Standards should be wanted.

The production of standards relies upon the willingness of all parties concerned to reach a voluntary agreement among themselves for one or more stated purposes.

2. Standards should be used.

Application of standards relies upon the voluntary commitment required in their preparation being extended to their use. The publication of a standard is of little value if it is not applied. The intended application of a standard should be clearly understood at the start and borne in mind throughout its preparation.

Standards should be written in a simple and clear way. Verification of compliance with specified requirements should always be possible within a realistic time and at a reasonable cost.

3. Standards should be planned.

The social and /or economic benefits of a standard should be compared with the total cost of preparing, publishing and maintaining it. The responsible committees should consider whether it is likely to be feasible to prepare the proposed standard in a technically and commercially acceptable form in time to be of use. In areas of rapid development, the balance should be struck between the risk of inhibiting innovation by premature standardisation and the danger of allowing the spread of divergent and mutually incompatible solutions to the same problem. If the latter occurs, the cost of subsequent standardisation is likely to be much greater.

A standard expresses what has been established or is about to be established. The process of writing standards is essentially one of selection. A standard can contain only what the interested parties are prepared to agree on at the time it is written. Thus, decisions are needed on when and how it is appropriate to standardise in a rapidly developing industry or to satisfy new community needs relating to safety or the environment.

Standards should be reviewed at regular intervals and appropriate action taken. A standard that does not evolve in keeping with changing circumstances or technological advances may become irrelevant or inhibit progress.

4. Standards should not be duplicated.

Standardisation can be pursued at different levels: by individuals, firms, associations, countries, regions such as Western Europe, and worldwide. For economy of total effort, a standard should logically be prepared at the broadest level consistent with meeting the needs of interested parties within an acceptable time-scale. The simultaneous preparation, at different levels, of standards on identical aspects of identical subjects should be avoided as far as practicable.

For the same reason, any standards body embarking on a new project should take account of existing standards on the same subject, from whatever source. Even an international, de facto standard, suitable for formal adoption, may already be found to exist. In this respect, the intended result of regional and international standardisation is the harmonization of different countries' national standards through standards being adopted that are identical with, or at least technically equivalent to, those in other countries.

1.4.2.2 Subject, aspect and level of standardisation

The terms subject, aspect and level have already been used in describing the aims and principles of standardisation [4]. The majority of standardisation subjects are material objects such as bolts and nuts, copper tubes, domestic appliances, dental instruments, etc. Besides these hundreds of subjects, there are a great many more abstract subjects such as limits and fits, grading or sampling of minerals, noise assessment. Also, there are letter and graphical symbols like electrical ones or those used to denote surface texture. Because there are so many various standardisation subjects it is convenient to group them together into "domains". A standardisation domain is a group of related subjects and the following are a few examples: engineering, packaging and transport, food, agriculture, textiles, and chemicals.

As a logical means of presenting standardisation problems, the concept of "Standardisation Space" was first proposed by Dr. Lal Verman [17]. In the standardisation space, as shown in Fig.1.2, subject, aspect and level constitute the three axes of reference. In this orthogonal system of three axes, denoting standardisation space subjects and domains are presented along the X-axis and,

since there are a very great number of standardisation subjects, for convenience only examples of domains are indicated on the figure.

A standardisation aspect is a group of requirements or conditions, which must be satisfied by a standardisation subject if that subject is to be regarded as conforming to a standard. There are many aspects and to name only a few of them: specification, analysis, testing, sampling and inspection, code of practice. These are presented along the Y-axis in Fig.1.2.

Standards can be promulgated at different levels, the four most important levels being [4]:

1. The international level

   Standards such as those of the ISO and IEC, resulted from cooperation and agreement between a large number of independent sovereign nations having common interests. Such standards are intended for worldwide use.

2. The regional level

   Standards initiated by a limited group of several independent nations, or by a regional standards body, for their mutual benefit. Examples of the latter are the European Standards Committees CEN and CENEL, the Pan American Standards Commission COPANT and the Eastern European Group CMEA.

3. The national level

   Standards promulgated after consulting a consensus of all the interests concerned in a country, through a national standards organization, which is recognized as the proper authority for the issue of such standards.

4. The company level

   Standards issued by an individual company (or in some cases, a group of companies), prepared by common agreement between various departments of the company for guiding its purchases, manufacture, sales and other operations. The levels of standardisation are presented along the Z-axis in Fig.1.2.

A standard may be regarded as a document containing a solution of a standardisation problem; and the problems, which may be concerned with one or more subjects, generally with several aspects and handled at a certain level,

will occupy a defined volume of standardisation space in Fig.1.2.

It is obvious that the standardisation space as described above cannot be taken as a mathematical space of either continuous or discrete variables; it is to be regarded merely as a convenient device to illustrate the three important attributes of standardisation problems.

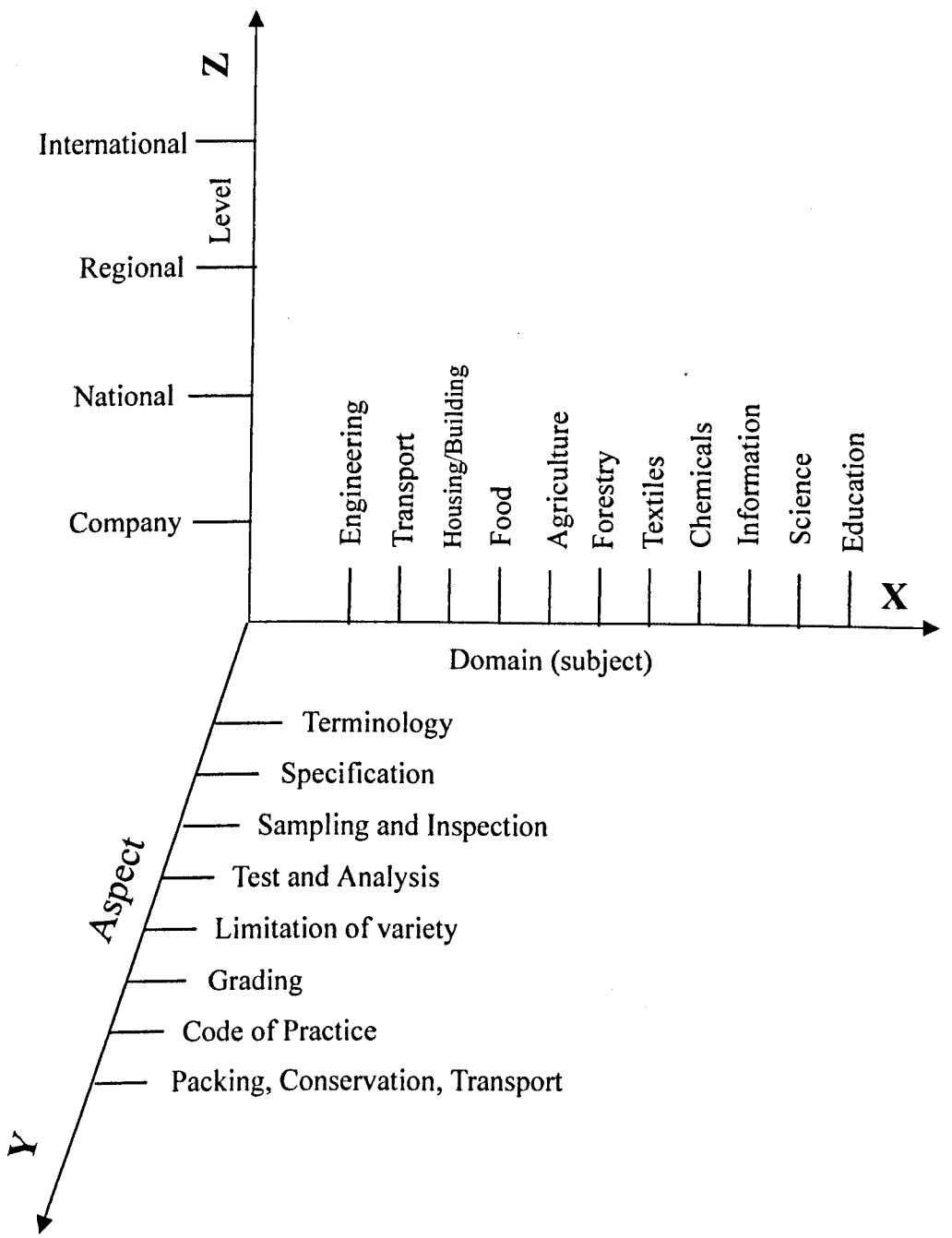


Fig.1.2 Diagrammatic representation of standardisation space (Sanders,1972)

1.4.3 Standards need in engineering ceramics

Standards are the international language of science and engineering. On the basis of standards, industrial suppliers and their customers around the world can reach assured understandings about products and their performance. With industrial markets rapidly becoming global markets, this language of science is more important than ever. It is especially important in highly technical applications.

For the engineering ceramics industry, the urgency is even greater. Many ceramic materials have only recently emerged from the laboratory. Yet, they must compete on equal terms with established materials such as metals and plastics. To do so, they must give users the assurance of quality and performance that is provided by standards.

1.4.3.1 The need for standards

There are many reasons to establish standards for engineering ceramics [18]. One is the creation of a common language. This will enable a manufacturer to communicate clearly with a customer's product engineers, designers, and purchasing agents anywhere in the world. Another need is to address concerns of public health and safety, where appropriate. This includes impact on the environment. Perhaps the primary need met by standards lies in the assurance they provide that a product meet requirements for quality and performance.

Then there are issues of compatibility. For some consumer electronics products, this is vital. A good example can be seen in the contest between VHS and Beta Max video tape. For engineering ceramics, this need may be less urgent but it must also be considered. There is also the issue of competitive materials. Purchasing agents and design engineers may be reluctant to specify an engineering ceramic for which no standards exist. They may instead turn to an alternate material or product that meets well-established standards.

Certainly, the development of standards will expand the base knowledge about the range of properties available in engineering ceramics. Simply having every participant speaking the same standard language will make the

pool of such knowledge grow.

With the rapid growth of international trade in engineering ceramics, the need for standards is particularly pressing. Standards are an important communications tool not only to industry but also to international affairs. Whether it is goods, services or knowledge, better communications underlie the ISO objective to encourage international exchange and mutual co-operation among the nations of the world. Standards constitute the language of international trade, cutting across ethnic and national borders.

A standard is a reference, an information item and at the same time a quality guarantee once the certificate of conformity has been obtained. In the absence of standards, every manufacturer develops his own labels, markers are often cut off from each other and competition slackens.

In time, the need for standards will probably increase substantially. Firstly, the ever wider of ceramic materials and the development of hybrid forms confront users, and notably small enterprises, with increasingly complex problems in their choice of material. It is only by drawing up suitable standards, and in particular by defining at least rudimentary classifications, purchasers can be helped to select materials and the commercialization of ceramic materials speeded up and expanded. Secondary, the increased stringency of quality requirements with regard to products calls for closer control of production processes (requisite degree of purity and controlled levels of impurities for components, for example). Greater use will therefore be made of quality control and non-destructive testing during fabrication, instead of testing products only at the end of the production line. Finally, broadening of the industrial base and progress in bio-medical research will continue to generate increased legislation on health safeguards and pollution control. If there are no economic incentives to use clean processes, we may expect to see increased recourse to standardisation.

1.4.3.2 The obstacles of standardisation

In most segments of the engineering ceramics market, it is clear that standardisation functions are still not being properly carried out, particularly because of the rapid changes in techniques and products.

There are many reasons to establish standards for engineering ceramics but there are also formidable obstacles to overcome. The numerous needs for standardisation are matched by an equally imposing list of obstacles to standardisation. One is the lack of a definition for engineering ceramics. Engineering ceramics must be distinguished from traditional or low-tech ceramics. Engineering ceramics usually require more sophisticated processing. And they often have designed-in microstructures to achieve certain properties. Such determinations are the work of a standards organization. But the many participants –materials producers, processors and manufacturers of derived products and consumers– make it more difficult to obtain a consensus on common standards.

Another obstacle to developing standards is the sheer variety of forms taken by these materials. Engineering ceramics can take the form of a powder, a coating, a fibre, a monolith, or a cellular honeycomb shape. Each may require its own set of standards, depending on the properties desired.

The range of applications is even broader. Engineering ceramics are widely chosen for mechanical uses, such as bearings and tools where hardness, strength, wear resistance, thermal resistance and safety is important. Another class of applications is based primarily on thermal and chemical properties. Ceramic engine components and heating elements are chosen largely for their thermal properties. This diversity of uses presents a problem for standards writers simply in the number of variations it requires. So the challenge is to find common terms and testing procedures that cover broad classes of materials and products.

The need for standards is most apparent when identical products are tested differently for the same property. Testing procedures for engineering ceramics today tend to be product-specific. One example is the wide range of tests used to measure particle size. There are many testing methods for this measurement, such as laser diffraction, Brownian motion, hydrodynamic chromotography or centrifugal sedimentation, but the standards for this measurement are usually established by each instrument maker, not by a standards organization.

Compressive strength is another property needed to test. In isostatic pressure testing at Corning, Inc., they use a pneumatic test. The sample is

placed inside an elastomeric boot and subject to measured stress by the application of pneumatic pressure. Another company tests the same parts by wrapping the sample in vinyl film and applying hydraulic pressure. However, a third company's test involves placing the parts in a hydraulic device that stresses them in only two dimensions, rather than three. In this case one is definitely needed, because test results today cannot be correlated.

Standardisation of engineering ceramics is still in its infancy. There are several reasons for this situation. Firstly, the data bases on these materials still contain too little information, since the fact that these materials have not been on the market for long makes it impossible to draw on experience, as in the case of conventional materials. Moreover, engineering ceramics are created in response to quite specific needs in terms of performances and characteristics. If they are to be taken into account, the measurement methods, tests and classification techniques in standardisation systems have to be adjusted. These adjustments are relatively slow compared with the engineering ceramics needs in the various countries because of the heterogeneity of the activities concerned, the amount of necessary pre-standardisation work and the many bodies involved (industrial federations, users, test laboratories, government authorities, individual firms, international organizations, etc.).

1.4.3.3 The approach to developing standards

The ISO/IEC Advisory Board on Technological Trends (ABTT) has proposed a three-phase approach to developing standards. The first step is to establish common terminology (definitions, nomenclature), units, symbols and abbreviations. This phase can be applied even to projects still in research and development. The second phase is characterization. As the product moves toward commercial status, work can begin on testing and measurement, evolving into process and production standards. The third phase is creation of product standards, to achieve interface capability and assured reliability.

In this sector, a considerable amount of pre-standardisation work has still to be completed so that standards and regulations can be drawn up and certificates of conformity subsequently issued. For this purpose, it is necessary:

—To agree on standardised test methods;

—To have all the validated information required on the properties of materials, concerning, for example, solidity, elasticity, density, fatigue strength, heat resistance, etc.;

—To assemble the necessary data on engineering design methods;

—To set up standardised data bases summing up the information and presenting it in a clearly intelligible form;

—To design expert systems which will enable users to master the new techniques and choose the best materials to meet their needs.

The Collyear Report in the United Kingdom stressed, some years ago, the importance of test methods standardisation. It also pointed to the need for database standardisation and for steps to ensure that available sources of materials data and design knowledge are widely publicized and made more readily available to industry. The setting up in 1987 of a Materials Information Centre in London as an easily accessible first point of contact for companies seeking sources of advice on materials can be seen as a response to the latter requirement.

Standardisation activities in the United Kingdom are directed along three main lines [1]:

(1) Standard test methods are developed, since manufacturers use these methods for monitoring the quality of the materials bought or, during the intermediate manufacturing phase, for non-destructive tests during operation, or for deciding on rejects at the end of a production line. In this area, the NPL is particularly active, often in conjunction with other bodies in the country, e.g. NEL or abroad.

(2) Standard specifications (performance standards) are produced to which the procedures to be adopted for conformity checking are often added. These performance standards define the requirements to be met, but without referring to a precise material or technology. Such work on specifications falls within the remit of NEL and to some extent NPL and is another important aspect of the national and international standardisation activities carried out.

(3) Regulatory codes (mandatory standards) are developed. These standards generally pool the results of practical experience and scientific research

and are presented in a form suitable for the industry concerned. In this activity NEL has a major role to play; it is to be noted that some balance with performance standards is desirable, for substitution may be prevented and innovation impeded in a sector regulated exclusively by means of mandatory standards.

Over and above the national programmes, standardisation processes obviously have an international role, if only to check the trends towards the fragmentation of markets and brake the protectionist tendencies they often conceal. Most of the national standardisation associations are affiliated with international organizations such as ISO. In the field of engineering ceramics, the speed-up in technological progress and the tremendous amount of pre-standardisation work required suggest, however, the need for specific initiatives.

The Versailles Advanced Materials and Standards (VAMAS) project is an attempt to fill this gap. This project, which was launched at the conference of the seven major countries in Versailles in 1982, is intended to:

— Promote co-operation on emerging technologies concerning advanced materials so as to encourage the use of joint mandatory standards for the manufacture of materials;

— Ensure the exchange of information on codes and specifications concerning materials and on the organizations in the different countries so as to facilitate co-operation and the adoption of joint standards.

Creating standards will not be easy. The field of engineering ceramics is continually changing. Product forms and applications vary widely. Even definitions and terms remain to be established. In general, it is very difficult to determine the best appropriate evaluating techniques for engineering ceramics when they are used as new applications. One realistic approach is to organize the groups of standards consisting of plural standards, in which standards also could be valuable for developing engineering ceramics [19].

Certainly there is far to go in achieving standards tests for properties, standard classifications of performance limits, and standard measurements of process parameters. It will not be easy to achieve these standards across the broad range of ceramic products and application. But it can be done if leading organizations make a commitment to do it.

1.4.4 Present situation of standardisation

The increasing use of ceramic materials for both structural and functional applications implies wide range commerce in powders and finished components. This market will necessarily require the availability of standards for measurement of powder properties as well as the determination of properties critical to the design, manufacture and performance prediction of ceramic parts.

Standardisation is an important policy not only for furthering the use of new materials but also for furthering the research and development which takes place in advance of it. In research and development, it is necessary to have standardisation and the common acceptance of methods of testing and evaluation and to have valid experimental data circulating smoothly at the international level.

Standardisation began on a national basis being one of the inevitable consequences of the Industrial Revolution. It started around the beginning of this century with almost simultaneous activity in the USA and Europe. The leader in the race was the American Society for Testing and Materials (ASTM), which was founded in 1898. Within Europe, the first official standards body was in the United Kingdom where in 1901, the original Engineering Standards Committee was formed as the forerunner of the British Standards Institution (BSI) [20].

The start of international standardisation was brought about by the electrical industry in the founding between 1904 and 1906 of the International Electrotechnical Commission (IEC). In April 1926, fourteen countries, which had then set up national standards bodies, met together to consider the extension of international collaboration to other fields. This resulted in the formation of the International Federation of the National Standardising Associations (ISA). The stresses and strains of the Second World War brought ISA activities to a half. From 1943 to 1947, it was temporarily replaced by the United Nations Standards Coordinating Committee (UNSCC), which had a membership of 18 nations. At the close of the war, the UNSCC decided that the time was ripe to create a new and permanent international body which could move forward and take over the work of international standardisation. A full-scale conference was held in London in October 1946, at which representatives of twenty-five

standards bodies agreed to set up the International Organization for Standardisation (ISO). ISO has proved to be a success and now has members comprising the national standards organizations of 120 countries. Padgett (1992) said the first country to standardise the engineering ceramics in their own right has been Japan. As early as 1981, they published JIS R1601 "Testing Method for Flexural Strength (Modulus of Rupture) of High Performance Ceramics." The JISC work concentrates on the standardisation of test methods and has an ongoing programme of 95 separate items, which are supervised by the Japan Fine Ceramics Association (JFCA).

BSI formed its engineering ceramics committee in 1985. Its initial terms of reference were to develop standard methods of tests, and had published several standards. Current work now included in the BSI work programme is the preparation of a glossary of terms.

The creation of the BSI committee was closely followed by the setting up of ASTM Committee C-28 on Advanced Ceramics in the USA. This committee has a comprehensive work programme, which is broken down into the following areas: Performance, Properties, Processing, Design & Evaluation and Characterization. ASTM has issued a standard "Test Method for Flexural Strength of Advanced Ceramics at Ambient Temperature," which is essentially an update of an earlier Military Standard MIL-STD-1942.

Other national standards organizations which are known to be active with regard to advanced ceramics include AFNOR (France) and DIN (FRG). Up until recently, progress with European Community countries has been monitored by a specially convened Ad Hoc Committee.

1.4.4.1 Standardisation activities in Japan

Industrial standards in Japan consist of Japanese Industrial Standards (JIS), which are decided by the government and of standards prepared by industrial associations, academic or technical societies, etc.

Under the Industrial Standardisation Law, JIS are enacted by the Minister of International Trade and Industry (MITI) after deliberation by the Japanese Industrial Standards Committee, or JISC. JISC has tweenty-nine divisional councils for each technical field, and the Divisional Council for Ceramics is in

charge of standardisation of engineering ceramics [21].

Chisaki [21] said that draft standards for JIS are provided through two channels: one is provided by related industrial organizations and technical societies (such as Japan Fine Ceramics Association) voluntarily or entrusted by the government and the other is that which the government creates. For standardisation of engineering ceramics, the former is primarily used.

Before 1980, most of the activities in the Divisional Council for Ceramics of JISC were standardisation of traditional ceramics such as refractories, pottery and chinaware. In the early 1980's, Japan's effort for standardisation of engineering ceramics began. The Ceramics Society of Japan (CSJ) compiled the draft of JIS on bending strength (JIS R1601-1981)at the request of the Agency of Industrial Science and Technology (AIST). It then drafted JIS pertaining to the modulus of elasticity, as well as, chemical analysis of silicon nitride and silicon carbide. CSJ has been recently working for standardisation of academic fields such as technical terms and chemical analysis.

From 1983, the Japan Fine Ceramics Association (JFCA), entrusted by the AIST, started research and development of full-scale standardisation for fine ceramics. Since then, JFCA has been in the center of the standardisation activities in Japan. In 1985, Japan Fine Ceramic Center (Foundation) was established. It started activities as a "research institute" which will study test procedures of fine ceramics and pre-standardisation research. In 1988, the AIST set up the Special Committee for Standardising New Materials within JISC. The committee compiled the "Recommendation for Promotion of Standardising New Materials–a basic guideline for standardisation of new materials" which selected 711 items to be standardised in ten years. Among them were 219 items related to fine ceramics.

In response to the above Recommendations, the Standardisation Committee of JFCA set up the following four expert committees as its substructure and began studies of standardisation for the field of fine ceramics.

JFCA-EC1 Thermal and mechanical characteristics

JFCA-EC2 Electric, magnetic and optical characteristics

JFCA-EC3 Powders, process and chemical characteristics

JFCA-EC4 Field of artificial materials for organisms

In 1991, EC5 was set up in addition to the above four expert committees for the purpose of promoting international standardisation activities on fine ceramics.

In July 1993, the Japan National Council for International Standardisation on Fine Ceramics (JNCISFC) was organized to support the task of secretariat of ISO/TC 206. JNCISFC set up two expert committees; the Supporting Committee for the secretariat of ISO/TC 206 (EC5) and the Domestic Representative Committee for ISO/TC 206 (EC6).

The Textile and Chemical Standards Division of Standards Department at the Agency of Industrial Science and Technology in MITI, along with the JFCA prepared the Standardisation Programme. Based on this programme, related organizations have been entrusted with standardisation studies and compiling JIS drafts.

As of July 1997, twenty-eight cases of enactment of JIS were achieved through efforts by such organization [22]. Concerning test methods and products standards of functional ceramics mostly focusing on electronic materials, the efforts of the Electronics Materials Industrial Manufacturer's Association of Japan and others led to the enactment of cases of JIS.

1.4.4.2 Standardisation research in the United States

Dapkunas (1992) said that standards research in the United States is conducted at several Government laboratories and private companies while academic institutions conduct more fundamental work which provides the understanding which underpins standards development. This research is generally coordinated by Government organizations and implemented through national and international standards bodies [23].

The American Society for Testing and Materials (ASTM) is the primary organization in which domestic laboratories participate in standards development and implementation in the United States. This is conducted through, primarily, the C28 committee, Advanced Ceramics. Specific topics are addressed in the subcommittee C28.01 Properties and Performance, C28.02 Design and Evaluation, C28.05 Processing and Characterization, C28.07

Ceramic Matrix Composites, C28.91 Nomenclature, and C28.94 ISO 206 TAG. As of July 1997, there are twenty-five standards developed by ASTM C28 committee [24].

To further the coordination of domestic standardisation research with international efforts, extensive participation occurs through the International Energy Agency (IEA) Annex II programme on ceramic standardisation. The Department of Energy (DOE) is the U.S. operating agent for these programmes and draws upon the expertise of the National Institute of Standards and Technology (NIST), the Oak Ridge National Laboratory (ORNL), and several industrial powder producers and component fabricators with an interest in the implementation of advanced ceramics.

The U.S. Department of Defense has traditionally recognized the importance of standards and pre-standards research, which culminated in the well-known Military Handbooks. Although the purpose of the work is the production of military hardware, research results are often of value to industry.

Two major government laboratories are currently engaged in the ceramics standards research field. The Oak Ridge National Laboratory, as the lead organization in the DOE Advanced Heat Engine Program, conducts extensive research on the mechanical behavior of heat engine ceramics. The NIST has traditionally led U.S. pre-standards research in response to its mission to aid industry in this field. In addition to the leadership of relevant ASTM C28 subcommittees, VAMAS TWA 3 and 14, and IEA Subtask 6 coordination, the NIST has pursued the development of test methodologies for wear and mechanical properties, powder characterization, the development of Standard Reference Materials (SRMs) and the development and distribution of evaluated databases of ceramic properties.

NIST, in cooperation with the Gas Research Institute, has developed a Structural Ceramics Database (SCD) which contains evaluated thermal and mechanical property data for several varieties of silicon nitride and silicon carbide. This database was issued by the NIST Standard Reference Data Programme in 1991 as SCD 1.0. NIST also distributed approximately 1000 different SRMs through its SRM Programme and has undertaken the development of several pertinent to the advanced ceramics community.

1.4.4.3 European standardisation activities

Padgett (1992) said that there has been much interest in the standardisation of advanced ceramics within Europe and within the EC in particular. This culminated in a mandated request from the Commission to both CEN and CENELEC for the establishment of a comprehensive programme for setting up European prestandards (ENV) and European Standards (EN) in the field of advanced industrial ceramics. It was further requested that the programme should be elaborated taking into account international standardisation activities [20].

The Technical Board of CEN acted very quickly and created a new Technical Committee CEN/TC 184 with the provisional title of "High Performance Ceramics." The Secretariat of the new TC was allocated to BSI. The inaugural meeting of CEN/TC 184 was held at the Manchester offices of BSI.

The title of TC 184 was confirmed as "Advanced Technical Ceramics" with its scope now being:

"Standardisation in the field of advanced technical ceramics with specific tasks being classification, terminology, sampling and methods of test. The methods of tests are to include physical, chemical, mechanical, thermal and textural properties for ceramic powders, monolithic ceramics, ceramic composites (including ceramic fibres and whiskers) and ceramic coatings."

CENELEC has also been responsive to the mandated request from the European Commission. They have held two meetings of experts from the various national bodies to determine whether a separate CENELEC work programme is required in the field of advanced technical ceramics. As a result the Technical Board of CENELEC has approved a standardisation programme in the field of advanced ceramics and has set up a new Task Force (BTTF 63-2) under British convenership. It is anticipated that its potential work might belong to the fields of interest of a number of technical committees of both IEC and CENELEC.

Of the eighteen CEN National Standards bodies, ten are now actively participating in the work of TC184. These represent France, Germany, Italy, Uk, Spain, Belgium, Netherlands, Switzerland, Sweden and Finland.

Morrell (1994) said that five working groups have been formed and have been operating under a CEC mandate for the development of an extensive series of premarket standards for advanced technical ceramics [25].

WG 1 Classification

WG 2 Ceramic Powders

WG 3 Monolithic Ceramics

WG 4 Ceramic Composites

WG 5 Ceramic Coatings

The detailed work programmes of working groups are listed in Appendix 1.

1.4.4.4 International standardisation activities

For the international standardisation of engineering ceramics, there was a collaborative research under VAMAS (Versailles Agreement on Advanced Materials and Standards) to verify test procedures for advanced ceramics prior to formal standardisation. The VAMAS project was launched at the conference of the seven major countries in Versailles in 1982. Morrell (1992) said that under the umbrella of VAMAS, three projects had been commenced which involve advanced ceramics. Technical Working Area (TWA) 1 was established to encourage pre-standardisation research of wear test methods, and has concentrated to a large extent on wear-resistant ceramics with inter-laboratory studies of test reproducibility. A number of these exercises have been conducted, and some standards have resulted from them. TWA 3, on advanced technical ceramics, was initiated to aid the worldwide push for improved testing standards, essential to improvement of the consumer's ability to understand ceramics, and to compare them on a consistent basis. TWA 14 was formed to address the issue of formal classification of advanced ceramics [26].

The International Energy Agency (IEA) Annex II programme on ceramic standardisation also provided a forum for the exchange of pre-standards research. The key activities of this progrmme are organized by subtask as follows: subtask 2, characterization of ceramic Powders; subtask 3, characterization of Dense Ceramics; subtask 4, Measurement of the Fracture Strength of Ceramics; Subtask 5, Flexural and Tensile Properties of Ceramics;

and Subtask 6, powder Characterization Continuation (of subtask 2).

On 20-21 April 1992, the International Conference on the promotion of standardisation for Fine Ceramics jointly supported by ISO was held in Nagoya, Japan. This conference was based on the statement regarding "advanced standardisation" made by the ABTT. Following the close of the conference, a "Nagoya Declaration" was issued, emphasizing the necessary of an early start to international standardisation activities, including the establishment of a new Technical Committee (TC) in ISO.

After the voting of member countries, the ISO/TC 206 for fine ceramics was officially established in November 1992 with 5 P-members (Participate members) and 21 O-members (Observer members). The ISO appointed Japan as Secretariat to this TC. Dr. Takashi Kanno and Dr. Samuel Schneider of NIST were nominated as Secretary and Chairman, respectively.

On 25 May 1994, the International Workshop on the Standardisation Activities for Fine Ceramics was held in Tokyo, Japan. The primary purpose of this workshop was to communize the present situation of fine ceramics standards in each country and to become acquainted with each other. Following this workshop, the first plenary meeting of ISO/TC 206 was held in the same place on 26- 27 May 1994.

As a starting point for the establishment of international standards for fine ceramics, the first plenary meeting of ISO/TC 206 have reached the following conclusions:

1. Title and scope of ISO/TC 206

   Title: Fine ceramics*

   * Alternative terms for fine ceramics are advanced ceramics, engineered ceramics, technical ceramics, or high performance ceramics.

   Scope: Standardisation in the field of fine ceramic materials and products in all forms: powders, monoliths, coatings and composites, intended for specific functional applications including mechanical, thermal, chemical, electrical, magnetic, optical and combinations thereof. The term "fine ceramics" is defined as "a highly engineered, high performance, predominantly nonmetallic,

inorganic material having specific functional attributes."

2. New work items for ISO/TC 206

   NWI 1 Test methods for particle size distribution of ceramic powders

   NWI 2 Test methods for flexural strength of monolithic ceramics at room temperature (RT)

   NWI 3 Test methods for hardness of monolithic ceramics at RT

   NWI 4 Classification of fine ceramics

3. Adoption of working groups and conveners

   ISO/TC 206 approved the formation of working groups with conveners, to address the work items given in ISO/TC 206 document number N24. The working groups and conveners are listed in ISO/TC 206 document N26.

4. Working groups and conveners for ISO/TC 206

   WG1 Particle size distribution of ceramic powders Japan.
   Mr. Jun-Ichiro Tsubaki, Japan Fine Ceramics Center

   WG2 Flexural strength of monolithic ceramics at RT USA.
   Mr. George Quinn, National Institute of Standards and Technology

   WG3 Hardness of monolithic ceramics at RT Japan.
   Mr. Syuuji Sakaguchi, National Industrial Research Institute of Nagoya

   WG4 Classification of fine ceramics

5. ISO/TC 206 requests that preferably a CEN member state becomes a P-member for the purpose of providing a convener for the draft work item on classification only, or alternatively through the Vienna Agreement between ISO and CEN that CEN/TC 184 convenes and drafts the work item on classification for parallel development and voting.

6. ISO/TC 206 approved a resolution that representatives from ISO/TC 206 and CEN/TC 184 should meet to discuss cooperation between the two

committees.

7. ISO/TC 206 approved the formation of an advisory group on planning as described in ISO/TC 206 document number N18.
8. ISO/TC 206 will establish a cooperation with CEN/TC 184 and liaisons with relevant ISO or IEC technical committees and other organizations such as VAMAS and International Ceramic Federation.

At the Second plenary meeting of ISO/TC 206 held in Kuala Lumpur, Malaysia on 1st and 2nd June 1995, an additional two working groups (WG5 and WG6) were organized to address additional new work items, i.e. NP5 and ISO/NP 15490, respectively.

At the third plenary meeting held in Cairns, Australia on 20th July 1996, additionally, four working groups (WG7, WG8, WG9 and PWI) were established corresponding to the approval of three new work item proposals and a preliminary work item.

The fourth plenary meeting of ISO/TC 206 was held in July 1997 in China. The 5th plenary and working group meeting was hosted by the Republic of Korea on 24-29 September 1998 as a satellite symposium of the 3rd International Meeting of Pacific Rim Ceramic Societies to be held on 20-23 September 1998 in Kyongju, Republic of Korea. The 6th plenary meeting and working group meetings was held in Canada in 1999.

The number and title of projects in development in ISO/TC 206 are listed in Appendix 2.

1.5 Summary

Engineering ceramics are ceramic materials used for engineering purposes. As compared to traditional ceramics that are made with firing of the natural inorganic material such as china and porcelain, glasses and cement, etc., engineering ceramics are made with artificial materials such as alumina, zirconia and silicon nitride. Therefore, engineering ceramics are possible to have excellent thermal, mechanical and chemical properties, according to control material, chemical composition and process.

The market growth of engineering ceramics seems to be much more

sluggish than was expected over the past 10 years. The obstacles to commercialization of engineering ceramics were reviewed. It was found that three major problems – Economics, Reliability and Applicability– would be tackled for more efficient development of the market. It is a fact that there are many instances where even the cost performance is higher when compared to materials now in use (mainly metals). Another obstacle to commercialization of engineering ceramics is the lack of reliability in such material properties related to failure or fracturing during service. Typically, these are strength and toughness. The reasons that applicability was considered as an obstacle to commercialization are the fact we are still uncertain about ceramics applications, our limited experience with this material, the absence of concrete steps to put ceramics into practical use, and the fact that methodology has not been established yet. To solve the aforementioned problems and strengthen promotion of commercialization, quality standards and standardisation of evaluation methods are the major issues proposed by the SAMPE Japan's Ceramics Committee.

There are many reasons to establish standards for engineering ceramics. One is the creation of a common language. This will enable a manufacturer to communicate clearly with a customer's product engineers, designers, and purchasing agents anywhere in the world. Another need is to address concerns of public health and safety, where appropriate. This includes impact on the environment. Perhaps the primary need met by standards lies in the assurance they provide that a product meet requirements for quality and performance. The need for standards is most apparent when identical products are tested differently for the same property. Testing procedures for engineering ceramics today tend to be product-specific.

The Collyear Report in the United Kingdom stressed, some years ago, the importance of test methods standardisation. It also pointed to the need for database standardisation and for steps to ensure that available sources of materials data and design knowledge are widely publicized and made more readily available to industry.

Creating standards will not be easy. The field of engineering ceramics is continually changing. Product forms and applications vary widely. Even definitions and terms remain to be established. Certainly it is far to go in

achieving standard tests for properties, standard classifications of performance limits, and standard measurements of process parameters. It will not be easy to achieve these standards across the broad range of ceramic products and application. But it can be done if leading organizations make a commitment to do it.

The present situation of standardisation in Japan, the United States, Europe and the international level were presented. The first country to standardize engineering ceramics has been Japan in 1981. BSI found its engineering ceramics committee in 1985. Its initial terms of reference were to develop standard methods of testing, and had published several standards. Current work now included in the BSI work programme is the preparation of a glossary of terms. ASTM committee C-28 on Advanced Ceramics in the USA has a comprehensive work programme, which is broken down into the following areas: Performance, Properties, Processing, Design & Evaluation and Characterization. The Technical Boards of CEN acted very quickly and created a new Technical Committee CEN/TC 184 with the title of Advanced Technical Ceramics. The ISO/TC 206 for fine ceramics was officially established in November 1992. A number of work projects have been conducted and some draft standards have resulted from them.

CHAPTER 2
Measurement of Flexural Strength

2.1 Introduction

In today's society there exists a vast, often invisible, infrastructure of services, supplies, transport and communication networks. Their existence is usually taken for granted but their presence and smooth operation are essential for everyday life. Part of this hidden infrastructure is the science of measurement –metrology.

Accurate measurements are required for the efficient manufacture of components for such varied things as internal combustion and gas turbine engines, where reliability and long life depend upon manufacturing tolerances of micrometers. In terms of high-technology industrial production, the list of applications requiring accurate measurement is endless. Measurement and measurement-related operation are estimated to account for between 3% and 6% of the GPD in industrialized nations [28].

The most important reason for measurement is to ensure a safe product. Some products, like bridges, airplane wings and ladders need to be measured for strength as a fundamental safety requirement. In the 19th century, failures of steam boilers and bridges caused public outrage which provided incentive for the emergent measurement science. Time has brought additional pressures for more rigorous measurement to be performed. Safety has become more important because the legal system's trend toward strict product liability makes product failure more costly, thus encouraging more measurement to prevent failure.

Some other products are not ordinarily thought of as a potential safety problem, but measurement may be important for other reasons, like assuring that customers will be satisfied with the product. Research and development laboratories also use standard measuring methods as a reliable way to compare

materials or products to determine the results of variations in formulation and processing.

The brittle nature of ceramics e.g. silicon is sometimes a benefit because failure is very clear. Very often with ductile materials some plastic flows can occur which is undetected e.g. creep or hysteresis. With ceramics if the system works then it is perfect – any ceramic failure is obvious. With ductile materials partial failure can go undetected and unvalidate results unknowingly.

Engineering ceramics have several structural applications, such as the turbine blades in hot engines, the nozzle in highly corrosive metallurgical processes, or the substrates for electronic insulation modules. In such applications ceramic materials must assume primary structural functions at high stress levels, even under dynamic loading. The mechanical properties of a material determine its limitation for structural applications where the material is required to sustain a load. Thus, adequate technological control of the mechanical properties of engineering ceramics has become issues of major importance.

Strength is one of the most important mechanical properties of engineering ceramics. The measurement of strength can be conducted in a number of different ways, such as uniaxial tensile strength, hydrostatic tensile, theta, three-point bending, four-point bending, uniaxial compressive and diametral compression [10].

Tensile strength testing is typically used for characterizing ductile metals. Ceramics are not normally characterized by tensile testing due to the high cost of test specimen fabrication and the requirement for extremely good alignment of the load train during testing. Any misalignment introduces bending and thus stress concentration at surface flaws, which results in uncertainty in the tensile strength measurement.

The strength of ceramic materials is generally measured by bend testing (also referred to as flexure testing). The strength characterization data for ceramics are reported in terms of modulus of rupture (MOR) or bend strength (also flexural strength).

Measurements of flexural strength must be accurate if they are to be really useful and reliable. The variability in results of measurement can have a negative effect on the advancement of ceramic materials, particularly those materials being used for structural applications. These fluctuations are a result of the inconsistency of the materials themselves, intrinsic scatter in the material, and at times, the measuring methods. To obtain accurate and consistent measuring methods and, thus, results, the amount of research in the area of measurement of mechanical properties for engineering ceramics has climbed steadily during the last decade.

In this chapter some suitable and commonly used measuring methods for flexural strength of engineering ceramics are briefly described. In addition, the statistical treatment of the variable strength data is demonstrated. Finally, the factors that would influence the strength are discussed.

2.2 Measuring methods for flexural strength

Flexure testing is one of the most traditional and common means to measure the strength of engineering ceramics. Several test techniques for flexure testing of engineering ceramics have been developed. These techniques can be grouped into two methods: uniaxial flexure tests and biaxial flexure tests. There are many similarities between them. Nevertheless, there are some differences that warrant attention.

2.2.1 Uniaxial flexure tests

The flexural strength of engineering ceramics is conventionally characterized by beam bending tests. The loading in beam bending tests is applied in a single direction and thus produce uniaxial stress field. The beam bending tests are therefore referred to as uniaxial flexure tests.

The specimen in beam bending tests can have a circular, square, or rectangular cross section and is uniform along the complete length. Such a beam specimen is much less expensive to fabricate than a tensile specimen. The rectangular section beam tested in bending is by far the most common

brittle material test specimen. The theoretical stresses in these beams are well known and the positions of load application, which are particularly important for small specimens, can be defined easily.

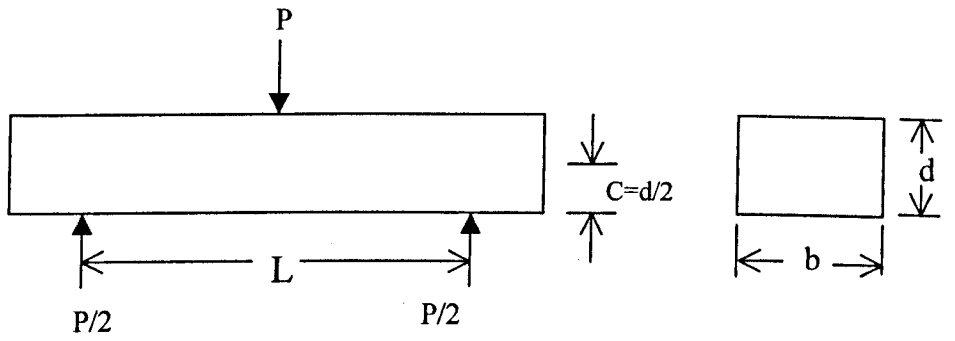
Beam bending tests can be conducted with the same kind of universal test machine used for tensile and compressive strength measurements. As shown in Fig. 2.1, the test specimen is simply supported and the load is applied either at the centre (three-point loading) or at two positions (four-point loading). The stress solution for the beam bending tests is known and well developed in the materials text books [29,30]. The flexural strength is defined as the maximum tensile stress at failure and is often referred to as the modulus of rupture, or MOR.

The four-point beam system assures a simple stress state which is easier to analyze than the relative complex biaxial stress state associated with the three-point beam specimen. Nevertheless, the three-point loaded beam system is preferred when investigating material or process development, because of smaller specimen size, or when attempting to pinpoint fracture origin location. On the other hand, the four-point loaded beam is preferred when determination of strength for design purposes is desired, because the centre span is uniaxially stressed, i.e., no shear stresses exist. It is concluded that each of these systems is suited for a particular application and each has different advantages and disadvantages.

The uniaxial test method for the measurement of flexural strength of engineering ceramics has been standardised by the JIS, ASTM, DIN and ANFOR. The test procedures and errors associated with beam test are more fully dealt with in Chapter 3. The standardisation activities and comparison of existing standards are estimated.

SIDE VIEW END VIEW

(a) 3-POINT



(b) 4-POINT

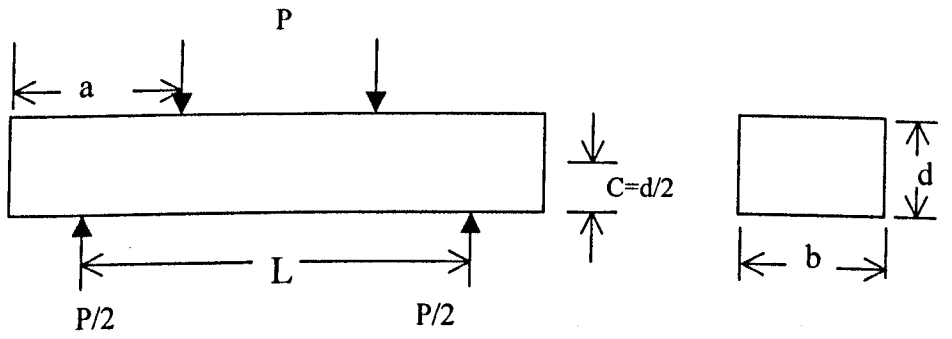


Fig. 2.1. Three- and four-point beam bending tests

2.2.2 Biaxial flexure tests

Uniaxial flexure tests, such as three- or four-point flexure of beam specimens, have long been used to measure ceramic strengths. This test method, or some variation of it is refined to allow for rocking of knife edges to accommodate warping of the specimen, is an important method because it allows measurement of strength on small bar-shaped specimens cut out of larger-shaped ceramic specimens. The measured strength depends, however, upon both the condition of the surface in tension and the condition of the edges in tension. The effect of surface condition on the strength of high alumina substrates has been studied by Gruszka, Mistler, and Runk [31] using transverse bending. In related experiments, Lo [32] has shown that large variations in strength, as measured in transverse bending, can result from differences in edge conditions with the same surface condition. It is difficult to separate edge and surface effects, so a second test method which would not be dependent upon edge condition is needed. In addition, uniaxial flexure tests may provide only a partial characterization of load-bearing capacity. In particular, there is currently no established method of relating biaxial to uniaxial strengths. There is a need for consideration of biaxial flexure tests since service applications of engineering ceramics generally involve multiaxial loads.

Watchman [33] has examined many possible ways for the biaxial flexure tests of ceramic substrates. This test method involves supporting a plate on three or more points near its periphery and equidistant from its centre and loading to a more central portion. The area of maximum tensile stress thus falls at the centre of the lower face of the plate and the strength should be independent of the condition of the edges of the plate. A number of variations of this technique exist as following:

1. The ring on ring (ring-loaded ring-supported, RL-RS) test

   The test involves supporting a circular plate on a ring and loading with a small concentric ring. An exact elastic analysis is available for the case of small deflection (less than the plate thickness). However, thin, high stress plates of materials such as chemically strengthened glass deflect as much as five times the plate thickness before failing, so the small deflection

elastic analysis is inadequate. It will gain considerable popularity now that the accurate numerical calculations of elastic stresses for large deflections have been carried out. ASTM Committee C-14 on Glass has developed a standard test method based on this technique.

2. The piston on ring (piston-loaded ring-supported, PL-RS) test

The test has been applied by Wilshaw [34] to measure the strength of polycrystalline alumina discs. He used a ball having a mechanical flat to apply the load. In this method, the piston loading is effectively a ring-load, as only line contact is obtained. A standard test method ASTM F324 has been developed based on this technique.

3. The ball on ring (ball-loaded ring-supported, BL-RS) test

The disc specimen uniformly supported on a circular ring is point-loaded by means of a smooth spherical ball. The test has been used in a study of the effect of surface condition on strength of ceramics by Mckinney and Herbert [35].

4. The piston on 3 ball (piston-loaded ball-supported, PL-BS) test

The test has been accurately analysed for small deflections. This method has an advantage over the preceding three method, in that support of the specimen on three balls allows the use of a slightly warped specimen. Thus, no surface grinding or polishing is required, in contrast to the ring supported techniques. The test has been adopted as an ASTM F394-78 standard for strength-testing ceramic substrates [36].

5. The ball on 3 ball (ball-loaded ball-supported, 4-Ball) test

The test involves a ball-loaded disc supported by three equi-spaced balls. It has a theoretical advantage over the piston on 3 ball test method in that there is no problem of assuring uniform loading over the surface of the piston. However, no exact elastic analysis for this case has been found. If an accurate stress equation for the 4-Ball test method is developed, it should be investigated further.

Although the ball-on-ring or 4-Ball test has several advantages, chief among them being its minimal requirements for test fixtures, the ball loading

generates steep stress gradients parallel to the specimen face and stresses only a small area of the disc specimen. For this reason, a hydraulic pressure loading fixture was constructed to provide a biaxial-tension-strength test in which the effective stressed area of the specimen is comparable to the conventional uniaxial-flexure test, such as four-point beam bend tests.

The hydraulic pressure loading test was apparently first used in the British glass industry for strength testing of plate glass. More recently, it has been used to evaluate residual strengths of glass and ceramic specimens following impact with liquid jets. In essence, the test consists of loading a disc specimen, which is supported along a concentric line support near its periphery, with lateral uniform pressure. An improved test fixture for biaxial-tension strength testing of ceramics featuring uniform pressure loading of discs was developed and qualified by D.K. Sheety et al. in 1983 [37]. In their work, biaxial data were obtained for an alumina ceramic, along with comparable uniaxial data from three-and four-point flexure tests.

In Chapter 4, three more attractive loading schemes that produce biaxial tension in flat-plate specimens, i.e. ring-on-ring, 4-Ball, and ball-on-ring tests will be dealt with more fully.

2.3 Statistical treatment of data

2.3.1 Introduction

Engineering ceramics are brittle materials and characterized by their complete lack of ductility i.e., under an applied load they show a wholly elastic behaviour until fracture occurs. Hence, high local stresses caused by loading attachments, misfits, misalignment, poor design or faulty manufacture cannot be relieved by plastic flow as in ductile materials. For this reason, brittle component designs differ from those for similar ductile components in that extra attention must be paid to detail, especially in highly stressed areas.

All engineering ceramics contain flaws such as cracks, pores, dislocations or inclusions, which result in stress concentration and fracture at a load well

below the theoretical strength. The strength of ceramic components is varied with the size of flaws within the structure. The flaw size depends on the characteristics of raw material, forming techniques etc. The strength of ceramic components is thus a statistical quantity. The design of brittle components must therefore be based on a probabilistic approach where the likelihood of failure of the component under a specified load can be estimated.

The distribution of fracture strength of engineering ceramics is commonly described by Weibull statistics [38]. The Weibull distribution function assumes that the brittle material fails as the weakest structural element fails, just as a chain breaks when the weakest link fails. In many cases of designs against brittle fracture, the Weibull statistics have been proved to be successful.

In the following sections, the statistical theory, Weibull distribution and the statistical treatment of the variable strength data necessary to explain the properties of engineering ceramics are developed.

2.3.2 Statistical theory

The variation in material strength due to the flaws in a particular material can be illustrated by fracture tests on a sample of specimens. A histogram of the fracture stresses of a set of nominally identical brittle test-specimens subjected to identical loading conditions (such as four-point bending) is shown in Fig. 2.2. The frequency of failure at a stress σ, (F_f), is defined as the fraction of the sample failing within the stress range σ to $\sigma + \delta\sigma$. In the limit, as the number of specimens (N) become large, the stress interval $\delta\sigma$ can be reduced to give a continuous distribution curve shown as the dotted line in Fig. 2.2.

For the analysis of fracture test data, it is more convenient to express the data in terms of the cumulative failure probability P_f, or alternatively, the probability of survival P_s. P_f can be defined as the fraction of the sample failing at or below a specified stress σ. In the limit, it is the integral of the frequency distribution with respect to stress.

$$P_f(\sigma) = \int_0^{\sigma} F_f \, d\sigma \qquad (2.1)$$

The probability of survival P_s is defined as $P_s = 1 - P_f$.

In practice, the cumulative failure probability is usually found from the data using the mean ranking approach. The N failure stresses of the sample are arranged in ascending order; the cumulative failure probability associated with the *i*th failure stress in the list is,

$$P_f(\sigma_i) = i/N+1 \qquad (2.2)$$

The probability distribution of the data can be plotted from this (see Fig. 2.3).

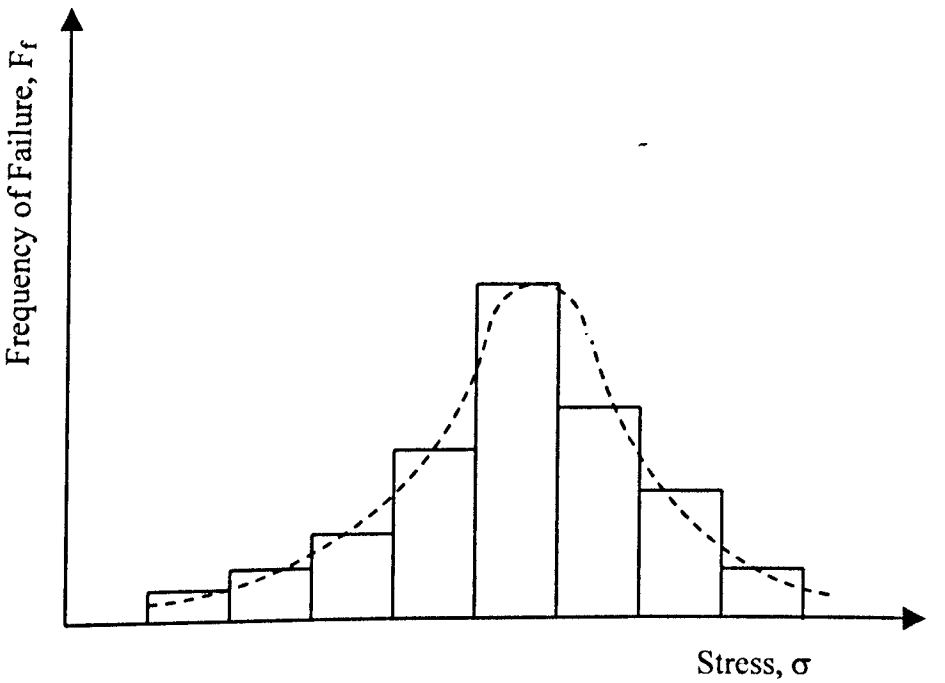


Fig. 2.2 Histogram of fracture stresses

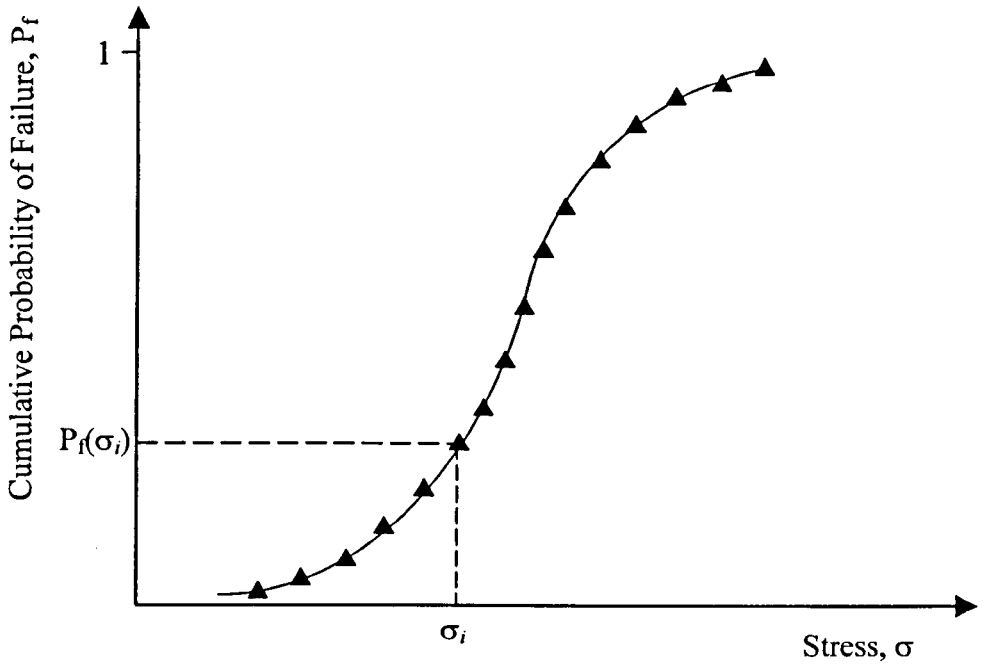


Fig. 2.3 Cumulative failures probability distribution

2.3.3 Weibull distribution

The Weibull cumulative failure probability distribution in analytical form may be written as [38],

$$P_f = 1 - \exp\{-[(\sigma - \sigma_u)/\sigma_o]^m\} \qquad (2.3)$$

Where m is the Weibull modulus

σ is the applied stress

σ_u is the threshold stress

and σ_o is a normalising factor

The Weibull modulus, m, is the exponent power to which the stress is raised in the Weibull expression. It is a measure of the consistency of the specimen failure stresses or an indication of the uniformity of the flaw distribution within the material. The normalised frequency of failure F_f is plotted in Fig. 2.4 against the stress ratio $\sigma/\overline{\sigma}$ for four m values. As can be seen in Fig. 2.4, high values of m are associated with more consistent material;

with an m of 20, the spread about the mean is seen to be relatively small, whereas, a great deal of scatter is observed with an m of 5.

The Weibull modulus is extremely dependent on the physical details of the material components, the method of manufacture and the quality of the surface finish. With such differences, it is impossible to associate a particular Weibull modulus with a particular type of material. Generally Weibull modulus for ceramic materials are found in the range $5< m<20$.

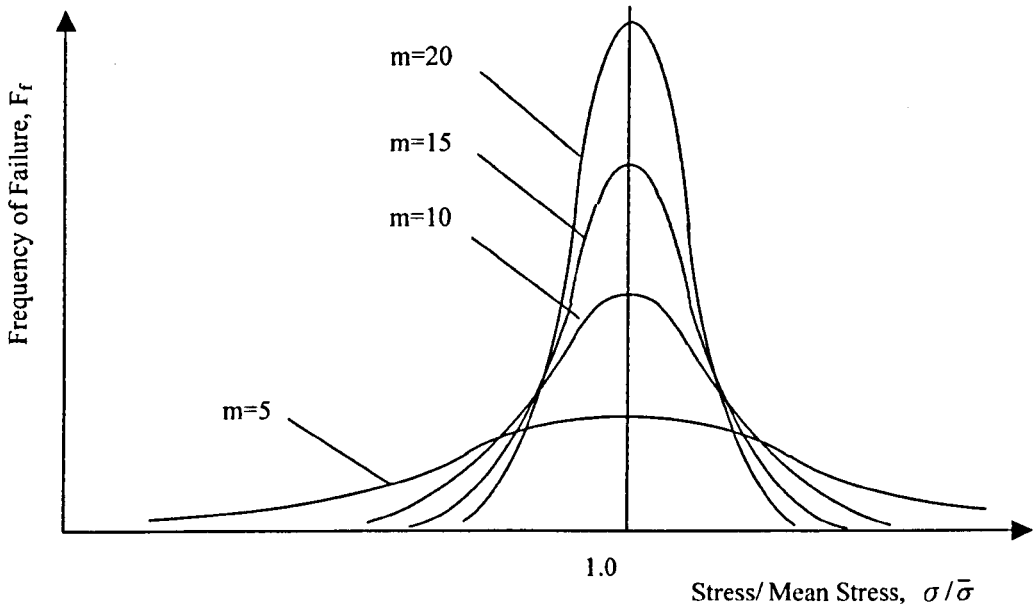


Fig. 2.4 Weibull frequency distribution for a range of m values

The threshold stress, σ_u, is the stress level below which the material will not fail. The failure probability is zero as the stress bellow σ_u. It is usually assumed that the value of σ_u is zero for the interpretation of fracture test data. The normalising factor σ_o has no simple physical significance. It has been shown [39] that σ_o is proportional to the mean fracture stress, $\bar{\sigma}_f$, of nominally identical specimens taken from an infinite population and that

$$\sigma_o = \bar{\sigma}_f / (\tfrac{1}{m}!) \qquad (2.4)$$

where (1/m)! denotes the gamma function. The gamma function is illustrated

as a function of m in Fig. 2.5.

Substituting for σ_0 in equation (2.3), it becomes

$$P_f = 1 - \exp\{-[(1/m)!]^m (\sigma / \bar{\sigma}_f)^m\} \quad (2.5)$$

Equation (2.5) is an important formulation of the Weibull distribution. The equation can also be expressed in terms of the median fracture stress, $\bar{\bar{\sigma}}_f$, of the distribution as [12],

$$P_f = 1 - \exp[-(\ln 2)(\sigma / \bar{\bar{\sigma}}_f)^m] \quad (2.6)$$

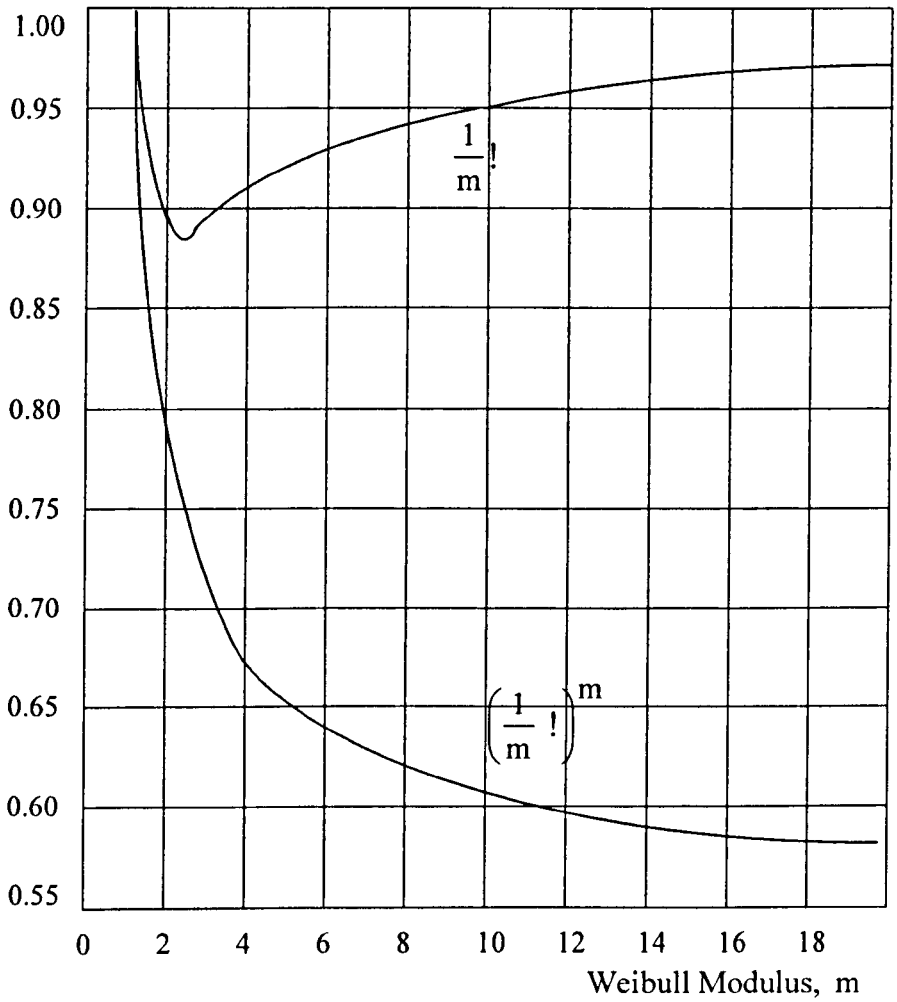


Fig. 2.5 The function (1/m!) and $(1/m!)^m$ versus Weibull modulus

2.3.4 The four-function Weibull distribution

The basic Weibull equation (2.5) applies only to nominally identical specimens in a particular batch subjected to a uniform uniaxial tensile stress. A general expression for the prediction of fracture behaviour of brittle components subjected to any kind of stress field has been developed by Stanley, Fessler and Sivill [40] in the form of a four non-dimensional parameters function.

As a consequence of the size-dependence of the strength of brittle components, it follows that the mean failure stress, $\overline{\sigma}_f$, for batches of different sized specimens, loaded under identical condition, will be different. Hence the mean failure stress, $\overline{\sigma}_f$, cannot be regarded as a material property. Instead a specific strength quantity, the mean strength of unit volume or unit area (i.e. the unit strength) should be used [39].

If the flaw distribution throughout the volume, V, of a uniaxial tensile specimen is statistically uniform, then the size effect relationship as,

$$\overline{\sigma}_f / \overline{\sigma}_{fv} = (v/V)^{1/m} \qquad (2.7)$$

where $\overline{\sigma}_f$ is the mean failure stress

$\overline{\sigma}_{fv}$ is the mean failure stress of a specimen of unit volume

v is the unit volume.

If a large sample of nominally identical specimens are tested under uniform uniaxial tensile stress, the basic Weibull equation (2.5) can be generalised by expressing $\overline{\sigma}$ in terms of $\overline{\sigma}_{fv}$. It follows that:

$$P_f = 1 - \exp\{-[(1/m)\,!\,]^m (\sigma/\overline{\sigma}_{fv})^m (V/v)\} \qquad (2.8)$$

where P_f is the cumulative failure probability of a specimen subjected to a uniaxial tensile stress, σ.

In the case where only surface flaws affect strength, the above is expressed in the same manner as follows,

$$\overline{\sigma}_f / \overline{\sigma}_{fa} = (a/A)^{1/m} \qquad (2.9)$$

where $\overline{\sigma}_{fa}$ is the mean failure stress of a specimen of unit surface area

a is the unit surface area

A is the surface area of a uniaxial tensile specimen.

and

$$P_f = 1 - \exp\{-[(1/m)\,!\,]^m (\sigma / \overline{\sigma}_{fa})^m (A/a)\} \qquad (2.10)$$

where P_f is the cumulative probability of failure corresponding to an applied uniaxial tensile stress, σ.

If a component is subjected to a non-uniform uniaxial stress, it can be regarded as an assembly of N elements, each so small that the stress acting on it is practically uniform. Since the weakest link concept assumes that failure in any one element results in failure of the whole component, then the survival probability of the component requires the survival of every element $(1-P_{fi})$. Since the survival probability of an element is independent of that of any other element in the component, the survival probability of the whole component is obtained from the normal statistical multiplication law for independent events, i.e.

$$(1-P_f) = (1-P_{f1}) \times (1-P_{f2}) \times \cdots\cdots (1-P_{fN}) \qquad (2.11)$$

Deriving the failure probability from equation (2.8) and allowing each element to become small it follows, in the limit, that the failure probability of the whole component becomes

$$P_f = 1 - \exp\{-[(1/m)!]^m (1/\overline{\sigma}_{fv})^m (1/v) \int \sigma^m \, dV\} \qquad (2.12)$$

where the integral is taken over the entire volume of the component .
Alternatively for the area formulation

$$P_f = 1 - \exp\{-[(1/m)!]^m (1/\overline{\sigma}_{fa})^m (1/a) \int \sigma^m \, dA\} \qquad (2.13)$$

where the integral is taken over the whole surface of the component.

In general the stress state at a point in a real component is not uniaxial but multiaxial; it is characterized by three principal stresses (σ_1, σ_2, σ_3). Equations (2.12) and (2.13) can be extended to cover multiaxial stressing by

introducing an independent action criterion [40] which assumes that the failure probability of an element due to any one principal stress is independent of the presence of any other principal stress.

The probability of failure of the component becomes:

$$P_f = 1 - \exp\{-[(1/m)\,!\,]^m (1/\overline{\sigma}_{fv})^m (1/v) \int (\sigma_1^m + \sigma_2^m + \sigma_3^m) dV\} \quad (2.14)$$

where the integral is again taken through the entire volume of the component,

and

$$P_f = 1 - \exp\{-[(1/m)\,!\,]^m (1/\overline{\sigma}_{fa})^m (1/a) \int (\sigma_1^m + \sigma_2^m + \sigma_3^m) dA\} \quad (2.15)$$

where the integral is taken over the whole surface of the component.

Further development of the equation is necessary when compressive stresses occur in the component, since the compressive strength of ceramic material far exceeds the tensile strength. If λ is defined as the numerical ratio of the compressive to tensile strengths, the general form of equation (2.14) may now be expressed with a Heaviside step function taking account of compressive stresses [40] as follows,

$$P_f = 1 - \exp\{-[(1/m)!]^m (1/\overline{\sigma}_{fv})^m (1/v) \int_V [(\sigma_1/H(\sigma_1))^m + (\sigma_2/H(\sigma_2))^m + (\sigma_3/H(\sigma_3))^m] dV\} \quad (2.16)$$

where $H(\sigma)$, the Heaviside step function, is defined by

$H(\sigma) = 1$ for tensile stress ($\sigma > 0$),
and $H(\sigma) = -\lambda$ for compressive stress ($\sigma < 0$).

Alternatively the area formulation becomes:

$$P_f = 1 - \exp\{-[(1/m)!]^m (1/\overline{\sigma}_{fa})^m (1/a) \int_A [(\sigma_1/H(\sigma_1))^m + (\sigma_2/H(\sigma_2))^m + (\sigma_3/H(\sigma_3))^m] dA\} \quad (2.17)$$

Equations (2.16) and (2.17) can be advantageously modified into a combination of non-dimensional factors. The three principal stresses are

represented as multiples of a suitable nominal stress, σ_{nom}, which is proportional to the load applied on the component, and the element volume is expressed as a multiple of the total component volume (V). Equation (2.16) becomes:

$$P_f=1-\exp\{-[(1/m)!]^m(\sigma_{nom}/\overline{\sigma}_{fv})^m(V/v)\Sigma(V)\} \quad (2.18)$$

where $\Sigma(V)$ is the stress volume integral given by,

$$\Sigma(V)=\int_V\{[\sigma_1/\sigma_{nom}H(\sigma_1)]^m+[\sigma_2/\sigma_{nom}H(\sigma_2)]^m + [\sigma_3/\sigma_{nom}H(\sigma_3)]^m\}dV/V \quad (2.19)$$

Likewise equation (2.17) becomes:

$$P_f=1-\exp\{-[(1/m)!]^m(\sigma_{nom}/\overline{\sigma}_{fa})^m(A/a)\Sigma(A)\} \quad (2.20)$$

where $\Sigma(A)$ is the stress area integral given by,

$$\Sigma(A)=\int_A\{[\sigma_1/\sigma_{nom}H(\sigma_1)]^m+[\sigma_2/\sigma_{nom}H(\sigma_2)]^m+ [\sigma_3/\sigma_{nom}H(\sigma_3)]^m\}dA/A \quad (2.21)$$

Equations (2.18) and (2.20) are the general "four-function Weibull equations" either of which give the failure probability of a component in terms of following four non-dimensional parameters [12]:

1. $[(1/m)!]^m$, the material consistency term, directly obtainable from Fig.2.5.
2. $(\sigma_{nom}/\overline{\sigma}_{fv})^m$, or alternatively $(\sigma_{nom}/\overline{\sigma}_{fa})^m$, the load strength factor where σ_{nom} is a nominal stress proportional to the load applied on the component.
3. V/v, or alternatively (A/a), the size factor. The volume of the component, V, is expressed in the same units of v.
4. $\Sigma(V)$, or alternatively $\Sigma(A)$, the stress-volume integral. After a stress analysis throughout the component volume, it can be determined for that particular shape of component under that particular form of loading, for a specified m value and choice of nominal stress.

A stress that can be easily calculated is chosen for the nominal stress; this is usually the maximum bending stress at fracture. If no simple expression for

the maximum bending stress exists, then a representative nominal stress, which is proportional to the load applied on the component can be used [13].

The normalised frequency of component failure, F_f, is obtained by differentiating failure probability, P_f, in equation (2.18) with respect to nominal stress. The mean nominal failure stress, $\overline{\sigma}_{nom}$, is then derived as the first moment of the frequency distribution about the frequency axis, i.e.

$$\overline{\sigma}_{nom} = \int \sigma_{nom} \cdot F_f \cdot d\,\sigma_{nom} \qquad (2.22)$$

It is shown [12] that equation (2.18) becomes:

$$\overline{\sigma}_{nom} = \overline{\sigma}_{fv}[v/V \Sigma (V)]^{1/m} \qquad (2.23)$$

Similarly equation (2.20) becomes:

$$\overline{\sigma}_{nom} = \overline{\sigma}_{fa}[a/A \Sigma (A)]^{1/m} \qquad (2.24)$$

It should be noted that the quantities required for the determination of $\overline{\sigma}_{nom}$ are precisely those required for the determination of the failure probability at a given nominal stress.

The standard deviation of the distribution, s, is obtained as the second moment of the frequency distribution about the mean nominal stress [12], i.e.

$$s = \int (\sigma_{nom} - \overline{\sigma}_{nom})^2 \cdot F_f \cdot d\,\sigma_{nom} \qquad (2.25)$$

Hence it follows that

$$s = \overline{\sigma}_{nom}\{[\,(2/m)!\,/\,(1/m)!^2\,] - 1\}^{1/2} \qquad (2.26)$$

The variance, v and the coefficient of variation are directly related to the standard deviation; the relationships are given in Table 2.1 for any set of N failure stresses.

The most important assumptions made in the derivation of the four-function Weibull equations (equations (2.18) and (2.20)) are [12]:

1. The material is isotropic and statistically homogeneous, i.e. the likelihood of finding a flaw of a given severity within an arbitrarily small volume, or

surface area, of the material is constant throughout the component.

2. The threshold stress is zero.
3. The weakest link theory applies, i.e. once a crack has initiated it will propagate without further increase in load, resulting in fracture.
4. A failure probability estimate applies to short term static loading; fatigue or creep is not considered.
5. The severity of a flaw is independent of its position in the component.
6. The three principal stresses acting at a general point contribute independently to the failure probability.
7. Compressive stresses can be simulated by the compressive to tensile strength ratio using the $H(\sigma)$ step function.
8. The unit tensile strength and the Weibull modulus are constant material properties.

The validity of these assumptions has been questioned. Sivill [12] suggested that with further development of the theory, some modification of the assumption may be called for.

Isotropy and statistical homogeneity (assumption 1) cannot be guaranteed. Marked anisotropy of material strength has been reported for hot-pressed Si_3N_4. Morrell [41] states that most ceramics do not have cubic crystal structures, resulting in nonequiaxed powder particles; most polycrystalline ceramics therefore have some degree of texture introduced by the powder-shaping process rather than completely random grain orientations; the distributions are usually nonhomogeneous and depend on the shape being processed; strength-limiting defects may be present either within the bulk or at the surface.

Assumption 2 implies that the Weibull postulate with zero threshold stress adequately described the variation of fracture strength of similar specimens. It seems unlikely that a statistical basis more satisfactory than the Weibull distribution will become available; a distribution incorporating a finite threshold stress may become worthwhile when larger batches are studied.

Assumption 3 describes the distinction between a brittle and a ductile material. Some modification of this assumption may be called for if the

materials contained fibre reinforcement. Assumption 4 is at present a limitation of the procedure; with further development of the theory it is probable that some allowance for a deterioration of unit strength with number of cycles (i.e. fatigue) or with time (i.e. creep) can be included.

Assumption 5 disregards the fact it is possible that flaws at the surface of a component may have more effect on the failure probability than similar flaws in the interior. With assumptions 6 and 7 the independent action failure criterion can be developed to handle multiaxial stressing, but these assumptions may result in underestimates of the stress-volume integrals computed from equation (2.19), though the error is probably only significant for components subject to marked biaxial stressing. No account is taken of the known weakening of the material with increased temperature in assumption 8, though it could probably be included in a modified four-function Weibull expression. Allowance for a temperature-dependent Weiull modulus may also be possible.

	From the data	In terms of the frequency	In terms of the Weibull Modulus
Standard deviation (s)	$\left[\frac{\sum_{i=1}^{N}(\sigma_i-\overline{\sigma_f})^2}{N-1}\right]^{\frac{1}{2}}$	$\left[\int_0^{\infty}(\sigma-\overline{\sigma_f})F_f d\sigma\right]^{\frac{1}{2}}$	$\overline{\sigma_f}\left[\frac{((2/m)!)}{((1/m)!)^2}-1\right]^{\frac{1}{2}}$
Variance (V)	s^2	s^2	$\overline{\sigma_f}^2\left[\frac{((2/m)!)}{((1/m)!)^2}-1\right]$
Coefficient of variation (C.O.V)	$s/\overline{\sigma_f}$	$s/\overline{\sigma_f}$	$\left[\frac{((2/m)!)}{((1/m)!)^2}-1\right]^{\frac{1}{2}}$

Table 2.1 Formulae for the standard deviation , variance and coefficient of variation.

2.3.5 Determination of material properties from fracture data

Two material properties, the Weibull modulus and the unit strength are required for a failure probability estimate from the four-function Weibull equations (2.18) and (2.20).

The Weibull modulus, m, is a material property whose value is a measure of the scatter in the material failure test data [12]. A linear, least-squares regression analysis is used in this work.

Flexure strengths within a sample were ranked in order and assigned a probability according to the formulae:

$$P=(i-0.5)/N \tag{2.27}$$

where P is the probability of failure , i is the ith specimen as the strength values are ranked in order, and N is the total number of specimens in the sample.

The strengths and probabilities were then graphed, where the abscissa is the natural log of stress, and the ordinate is $lnln(1/(1-P_f))$. A simple least-squares regression line is applied. The Weibull modulus is the slope of the line. Thus, the Weibull modulus can be readily and visually interpreted on a Weibull graph. This representation of the data is commonly used by engineers and scientists because of its simplicity and ease of interpretation [42].

The unit strength can be calculated from the mean nominal stress formulation (equation (2.23) or (2.24)). The unit volume tensile strength ($\overline{\sigma}_{fv}$) is then given by:

$$\overline{\sigma}_{fv}=\overline{\sigma}_{nom}\,[\,V\Sigma(V)/v]^{1/m} \tag{2.28}$$

The nominal stress ($\overline{\sigma}_{nom}$) chosen was the maximum bending stress at fracture. Hence $\overline{\sigma}_{nom}$ in equation (2.28) was the mean fracture stress ($\overline{\sigma}_{f}$) for each piece of specimen. Having determined the Weibull modulus of the material and knowing the stress distribution in the specimen, the stress-volume integral ($\Sigma(V)$) can be evaluated on the basis of the chosen nominal stress.

In a similar manner the unit area tensile strength ($\overline{\sigma}_{fa}$) is given by:

$$\bar{\sigma}_{fa}=\bar{\sigma}_{nom}[\, A\,\Sigma(A)/a]^{1/m} \qquad (2.29)$$

The accuracy of any failure probability estimate depends on the accuracy of the estimated material properties from the fracture testing of the sample material and therefore indirectly on the number of sample specimens considered. A complete analysis of the standard errors of material properties estimated from fracture tests is given by Sivill [12]. For Weibull modulus estimation from a sample of N specimens the true Weibull modulus has a 68% chance of falling in the range $m_{estimate} \pm \Delta m$, where Δm is the standard error in Weibull modulus estimation given by:

$$\Delta m \fallingdotseq m/\sqrt{2N} \qquad (2.30)$$

The accuracy of the mean nominal failure stress may be stated in a similar manner. The standard error of the mean nominal failure stress ($\Delta\bar{\sigma}_{nom}$) of a set of N specimens is given by:

$$\Delta\bar{\sigma}_{nom} \fallingdotseq 1.2\,\bar{\sigma}_{nom}/m\sqrt{N} \qquad (2.31)$$

The error in the unit strength is caused by the combined effects of the sampling errors on m and on the mean nominal failure stress of the specimens. If the stress in the specimen is known precisely and the specimen dimensions are considered free of error the standard error of the unit volume strength ($\Delta\bar{\sigma}_{fv}$) is given by:

$$(\Delta\bar{\sigma}_{fv})^2=[\Delta\bar{\sigma}_{nom}\cdot\partial\bar{\sigma}_{fv}/\partial\bar{\sigma}_{nom}]^2+[\Delta m\cdot\partial\bar{\sigma}_{fv}/\partial m]^2 \qquad (2.32)$$

The previously approximations for the standard error in the mean nominal failure stress (equation (2.31)) and the standard error in the Weibull modulus (equation (2.32)) to give:

$$\Delta\bar{\sigma}_{fv} \fallingdotseq \bar{\sigma}_{fv}\cdot 1/(m\sqrt{2N})\cdot\{\,2.88+[\,m/\Sigma(V)\cdot\partial\Sigma(V)/\partial m - ln(V\,\Sigma(V)/v)\,]^2\}^{1/2} \qquad (2.33)$$

In the same way the corresponding standard error in the unit area strength ($\Delta\bar{\sigma}_{fa}$) can be derived as,

$$\Delta \overline{\sigma}_{fa} \fallingdotseq \overline{\sigma}_{fa} \cdot 1 / (m\sqrt{2N}) \cdot \{ 2.88 + [m / \Sigma(A) \cdot \partial \Sigma(A) / \partial m - ln(A \Sigma(A)/a)]^2 \}^{1/2} \quad (2.34)$$

2.4 Factors influencing the strength

Accurate evaluation of the mechanical properties of ceramic material is very important when using the material for structural parts. Quinn [42] found that the variability in flexural strength results has several causes. The variability is often a consequence of the inherent scatter in tensile strength of brittle ceramics, but it is compounded by experimental errors in strength test methods and often is inconsistent in the materials themselves. Engineering Ceramics for structural applications must not only be strong, but have reproducible strength, from batch to batch and from day to day.

2.4.1 Material character

Even if engineering ceramics are made consistent, there still will be an intrinsic strength variability, a consequence of the Griffith-like behavior of flaws. Otherwise, it has been found that the average flexural strength of ceramics varies with the processing method and type of material.

2.4.1.1 Flaw size distribution

The presence of a flaw such as a crack, pore, or inclusion in a ceramic material results in stress concentration. The largest, most severely stressed, most sharp defect, or, in other words, the flaw with the highest stress intensity in a component, will initiate fracture. The natural variability in flaw size, location, and severity assures an inherent scatter of strength that is as much a measure of the material character as is the average strength.

Morrell [2,41] discussed this problem that monolithic engineering ceramics are brittle materials and typically show little, if any, plastic strain before failure over most of their useful range of operational temperature. Failure occurs by the catastrophic propagation of a crack-like defect when subjected to a sufficiently high stress level. This process has been extensively

modeled in the literature by either the Griffith fracture criterion or fracture mechanics analysis. These models show that the strength-limiting defects in engineering ceramics are of the order of 5-200 μm in size. This is in contrast with tougher metallic materials in which defects may be several millimeters or more in size without adversely affecting bulk properties. The small size of strength-limiting defects in ceramics means that they are difficult both to detect and to control. They may not even be present at the critical size in unstressed material, but may grow subcritically under load before failure. The worst defect will vary from specimen to specimen, and, consequently, the strengths of nominally identical specimens show a considerable spread of values, frequently with a coefficient of variation about the mean in excess of 10%.

Richerson[10] states that the defects which result in stress concentration and fracture at a load well below the theoretical strength can be a fabrication flaw or structural flaw such as a crack, pore, or inclusion in a ceramic material. The effect of a planar elliptical crack at the surface of a ceramic specimen is the easiest to analyze. This type of crack results commonly from machining, but can also occur due to impact, thermal shock, glaze crazing, or a number of other causes. The effect of three-dimensional flaws such as pores and inclusions have not been analyzed as rigorously. However, it is evident that the severity of strength reduction is affected by a combination of factors:

1. The shape of a pore.
2. The presence of cracks or grain boundary cusps adjacent to a pore.
3. The distance between pores and between a pore and the surface.
4. The size and shape of an inclusion.
5. The differences in elastic moduli and coefficients of thermal expansion between the inclusion and the matrix.

The variability in strength can be statistically analyzed using either an arbitrary strength distribution or a distribution based upon a flaw-size distribution coupled with a failure criterion. The most commonly used method of characterizing the spread of individual results is to apply Weibull statistical theory demonstrated in section 2.3.

2.4.1.2 Processing method

The room temperature strength of a commercial alumina ceramic has been characterized as functions of green shaping of the material. A series of tests on a 95% alumina ceramic (Sintox FA, Smiths Industries Ltd, U.K) conducted by Hanney and Morrell [43] demonstrated that average flexural strength varies with the manufacturing route. Small rods die-pressed to length and tested as rods appear relatively strong, but after correction are of similar strength to as-received bars derived from die-pressed tiles. With a ground surface they are weaker than die-pressed tiles. This result correlates with a lower fired density. Isostatically- pressed material with a surface ground finish appeared slightly stronger than die-pressed tile material even though it had significantly greater porosity. Slip-cast material, made from a slightly different kind of mix, was significantly stronger than die-pressed material in the ground condition. The extruded material proved to be more than 50% stronger than die-pressed material in the as-fired state even after correction for area or volume effects, but had a relatively low Weibull modulus. These tests on extruded material illustrated that the extrusion process is beneficial in generally improving the strength of the fired product over that of material produced by spray-dried powder routes, but that the strength achieved depends on extrusion conditions. It appears that the best properties are achieved in small scale extrusions where the size reduction on passing through the die, and hence the work done on the extrudate, is greatest [43].

2.4.1.3 Type of material

The amount of strength decreases resulting from grinding are different for various materials. Silicon carbide is more sensitive to surface flaws than silicon nitride. In G. Spur and T.H. Tio's study [44], it was concluded that the effect of increasing the grinding feed velocity on the component strength is dependent on the type of material being investigated. For silicon infiltrated silicon carbide (Si-SiC) an increase of feed velocity from 10 m/min to 20 m/min results in a strength reduction of approximately 10%. A similar increase of cutting speed when using hot pressed silicon nitride (HPSN) was found to have only a negligible influence on the strength.

A similar analysis for the correlation between the surface finish and the mechanical properties of commercial aluminas was carried out by L. Esposito et al. [45]. The surface roughness, microstructure and flexural strength of two commercial alumina ceramics with two different grinding procedures, longitudinal and planetary, were determined. The experimental results showed that the two alumina materials investigated had very different responses to the grinding procedures chosen. For the coarse-grained and pure alumina, the planetary grinding procedure caused an increase in flexural strength, about 13%, while for the less pure and fine grained alumina, the mechanical strength was only slightly affected by the two grinding procedures.

2.4.2 Test procedures

Another factor leading to inconsistent results is that of experimental error in the flexure test procedures. It is recognized that flexural strength of a group of test specimens is influenced by several parameters associated with the test procedure. Such parameters include the specimen size, surface finish, fixture geometry, loading rate, and sample size. These parameters are discussed individually in the following paragraphs.

2.4.2.1 Specimen size

For strength evaluation of brittle materials such as ceramics, Weibull analysis, based on the weakest link theory, which is related to unstable growth of Griffith flaws, is generally used. The Weibull analysis leads to a dependency of strength upon the size of the component or specimen [46]. This is a consequence of the greater likelihood of finding a large defect in the larger component.

An experimental investigation of strength-size relations in ceramic materials was conducted by Bansal et al. [47]. Bend strengths of two sizes of specimens of an alumina ceramic were determined under conditions that either enhanced or restricted subcritical crack growth. The specimen sizes differed in each linear dimension by a factor of five and the effective size of small specimens was varied by the use of both three- and four-point bending. The experimental results showed that the strength was dependent on specimen size

under both crack-growth conditions, and the dependence was associated with variations in flaws at fracture origins.

In a similar investigation of a commercial glass-ceramic (Pyroceram 9606, Corning Glass Works, Corning, N.Y.), Bansal et al. [48] also found that the fractures in this glass-ceramic were initiated most often at extrinsic flaws introduced during finish grinding of the specimens. Strengths of specimens failing from these surface origins did not exhibit size dependence, regardless of whether conditions restricted or enhanced subcritical crack growth. When subcritical crack growth was restricted, however, fracture sometimes initiated at a subsurface pore and a lower strength resulted. Because the probability of pore origins increased with specimen size, larger specimens tested under this crack-growth condition exhibited lower average strengths.

The equation widely used to express the size effect of ceramics is obtained by employing Weibull distribution function. When the average strength and effective volume of specimens with different sizes are denoted by σ_1 and σ_2, and V_{E1} and V_{E2}, and further Weibull modulus as m, the following equation is established:

$$\sigma_1 / \sigma_2 = (V_{E2} / V_{E1})^{1/m} \qquad (2.35)$$

In the case where only surface defects affect strength, the above is expressed in the same manner as follows denoting effective area as A_{E1} and A_{E2}:

$$\sigma_1 / \sigma_2 = (A_{E2} / A_{E1})^{1/m} \qquad (2.36)$$

Each of equations (2.35) and (2.36) represents the strength size effect of specimens failing from simple fracture origins. Different flaw populations will have different strength distributions associated with them and will scale in size differently. The presence of more than one flaw type is very common in engineering ceramics, and it will seriously complicate analyses to scale ceramic strength with size. A number of investigations [2,47,49] have identified and analytically modeled multiple flaw populations in engineering ceramics and will not be discussed in detail here.

Equation (2.35) indicates that there is a simple correlation between σ and

V_E; if $\log \sigma$ is plotted vs $\log V_E$, the data points should fall on straight line with slope $-(1/m)$. This relation was examined by Katayama and Hattori [50] with measuring the strength of sintered silicon nitride specimens of varied sizes in three-point, four-point bending and expanded-ring tests; good agreement between theory and experiment was obtained. The results thus suggest that mean strengths obtained under different conditions can be evaluated on the basis of effective volumes.

The strength size effect of ceramics was studied mainly with specimens and material components having larger volume than a standard specimen. Otherwise, Miyazaki et al. [51] proposed a strength evaluation method using a miniature ceramic specimen. The strength of specimens miniaturized to several levels was measured to investigate the size effect of strength. As a result, the measured strength of Al_2O_3 using miniature specimens increased with a decrease in specimen size, and this tendency was found to agree relatively well with the increase tendency of strength obtained from Weibull size scaling to use effective area.

2.4.2.2 Surface finish

It is known that the strength of ceramics depends on the surface roughness and the machining process of the test piece. Sometimes a specimen with a rough surface fails at a stress far lower than the essential strength of the material. Therefore, in the machining of ceramic parts as well as in the evaluating of strength, the relation of the strength of the part and the machining process is very important.

The effect of surface finish on the strength of the specimen is often expressed by the relation of the flexural strength and the roughness. Finely finished specimens give the highest and most constant values in strength tests. But roughly finished specimens, those which have more than several μm of roughness, give lower values compared to the former, and the strength decreases to almost half the original value with a lowering of the roughness.

The decrease of strength with lowering of the roughness is due to the occurrence of surface flaws from the machining process. In many applications,

machining may cause a variation in the surface and volume of Weibull parameters, as inferred by the work of Allor and Baker [52]. In Allor and Baker's study, grinding parameters caused as much as a 38% reduction in the characteristic strength of test bars. Since the bulk material properties were not affected this means that the surface Weibull parameters were significantly reduced.

It does not always follow, however, that a decrease in strength depends on the surface roughness. Surface flaws are considered to have directional qualities for the machining direction, and the flaw size is affected by the machining condition. The relation of the grinding condition and the bending strength in hot-pressed silicon nitride was investigated by Ito and Okuda [53]. They found that the strength properties of bending test pieces, the surfaces of which are finished parallel to specimen length and perpendicular to length by diamond grinding wheels of 400 and 200 mesh, are very different from one another for the same material.

An experimental study has been made by R.L. Allor et al. [54] to determine the influence of machining on fracture strength (four-point bend) of two potential turbine ceramics, namely sintered α-SiC and hot-pressed Si_3N_4. Machining aspects involve (a) types of cutting such as (i) use of a rotary table with a horizontal spindle, (ii) use of a rotary table with a vertical spindle, (iii) lengthwise, and (iv) transverse; (b) diamond wheel grit-size effects; and (c) downfeed effects in one machining pass. The experimental results showed that machining with metal-bonded diamond wheels may significantly reduce the strength of hot-pressed Si_3N_4, compared with machining with resinoid-bonded wheels; strength was reduced significantly by transverse grinding and by grinding with use of a rotary table with a vertical spindle.

A similar investigation of the influence of surface finish on the mechanical properties of advanced engineering ceramics was conducted by G. Spur and T.H. Tio [44]. They found that the direction of grinding has a considerable influence on the component strength. The ground component exhibits anisotropic properties whereby the tensile strength in a direction normal to the lay of the ground surface can be more than 30% lower than that parallel to its

lay.

In Hanney and Morrell's study [43], it was also found that the average flexural strength varies with the method of surface preparation. Machining with fine grit diamond wheels, or diamond lapping results in higher flexural strength than in as-fired or coarse grit machined material. It has been directly demonstrated that machining results in a surface compressive layer which is removed by annealing. Annealing also causes a loss in strength.

2.4.2.3 Fixture geometry

The measured strength will vary significantly depending on the fixture geometry. Richerson [10] presented an example, illustrating the magnitude and reason for this variation. For hot-pressed Si_3N_4 (Norton Company NC-132) specimens having a rectangular cross section of 3.2 $\times$6.4 mm, three-point bend testing over a 38mm span resulted in an average bend strength of about 930 MPa. Four-point bend testing of bars from the same batch resulted in an average bend strength of only 724 MPa. Uniaxial tensile testing of a comparable cross section of the same hot-pressed Si_3N_4 yielded a strength of only 552 MPa.

The stress distribution for three-point bending is shown in Fig. 2.6a. The peak stress occurs only along a single line on the surface of the test bar opposite the point of loading. The stress decreases linearly along the length of the bar and into the thickness of the bar, reaching zero at the bottom supports and at the neutral axis, respectively. The probability of the largest flaw in the specimen being at the surface along the line of peak stress is very low. Therefore, the specimen will either fracture at a flaw smaller than the largest flaw or in a region of lower stress. Three-point bend testing results in synthetically high strength values. Their values can be used for design only if treated statistically on probabilistically.

Four-point bend testing results in lower strength values for a given ceramic material than does three-point bending. The approximate stress distribution for four-point bending is shown in Fig. 2.6b. The peak stress is present over the area of the tensile face between the load point. The tensile stress decreases

linearly from the surface to zero at the neutral axis and from the load points to zero at the bottom supports. The area and volume under peak tensile stress or near peak tensile stress is much greater for four-point bending than for three-point bending, and thus the probability of a larger flaw being exposed to high stress is increased. As a result, the bend strength measured in four-point is lower than that measured in three-point.

Uniaxial tensile strength testing results in lower strength values for a given ceramic than does bend testing. Fig. 2.6c shows the stress distribution for uniaxial tension. The complete volume of the gauge section of a tensile test specimen is exposed to the peak stress. Therefore, the largest flaw in this volume will be the critical flaw and result in fracture when the critical stress is reached.

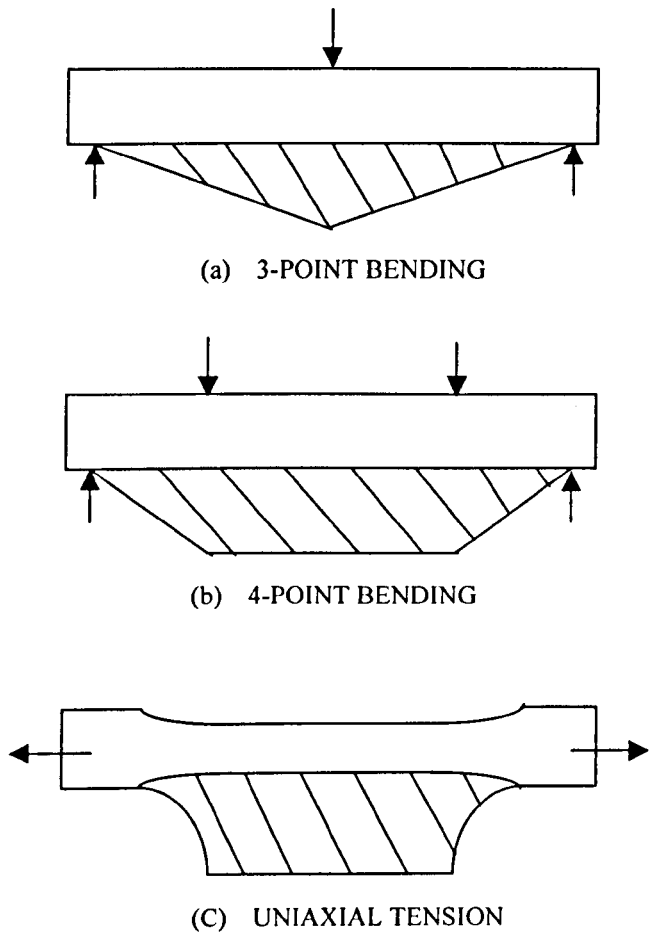


Fig.2.6 Stress distribution for bending and uniaxial tension (adapted from Refs 10)

Biaxial loading frequently occurs at the contact zone between two ceramic parts or between a ceramic and a metal part, especially during relative motion due to mechanical sliding or thermal cycling. Under certain conditions, very localized surface tensile stresses much higher than the applied load can result [10,55]. This is illustrated in Fig. 2.7.

Application of only a normal force N results in compressive stresses. Simultaneous application of a tangential force T results in localized tensile stress at the edge of the contact zone opposite the direction of the tangential force. This tensile stress is a maximum at the surface of the ceramic and rapidly decreases inward from the surface. The magnitude of the tensile stress increases as the coefficient of friction increases. It reaches a peak when the static friction is highest, but is immediately reduced once sliding begins because the dynamic coefficient of friction is lower.

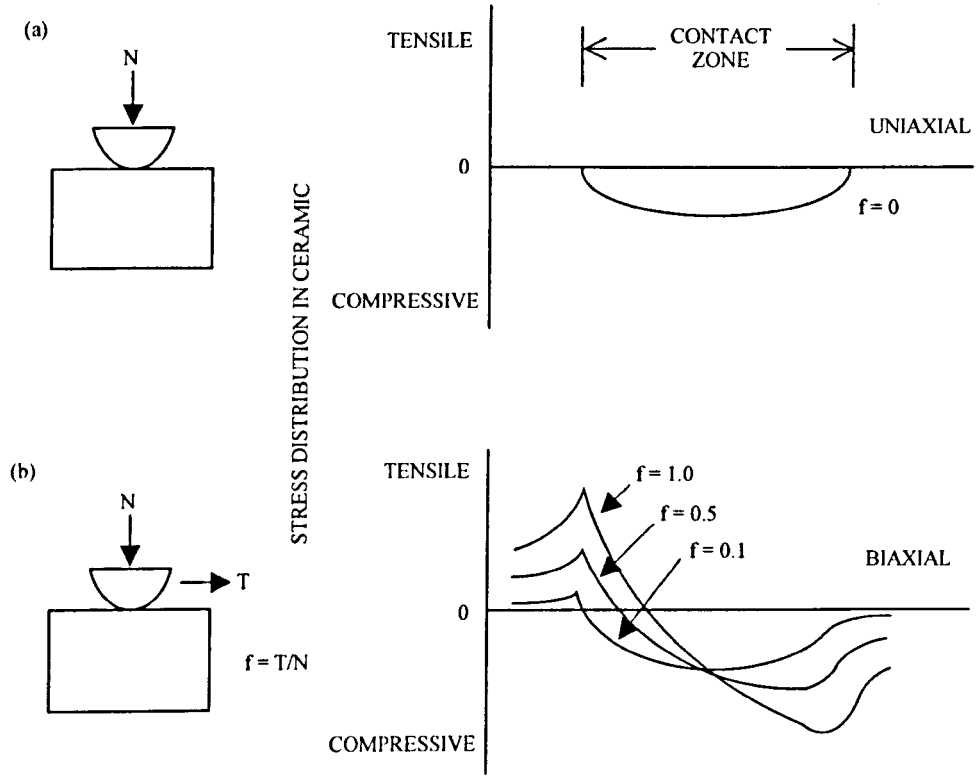


Fig. 2.7 Contact loading showing uniaxial and biaxaial effects on stress distribution. (Adapted from Refs 55)

2.4.2.4 Loading rate

The rate of loading can have an effect on flexural strength as the result of stress corrosion mechanisms, particularly at low strain rates. In general, the slower the rate of loading, the greater the opportunity for stress corrosion phenomena to weaken the specimen. Thus, fast loading rates are usually used in strength tests to minimize time dependent phenomena. The recorded time for failure for typical ceramics will range from 3 to 30 seconds.

An experimental investigation of the loading rate dependence of fracture strength in a reaction-sintered mullite ceramic was conducted by Wang et al. [56]. The variation in three-point bending strength as a function of loading rate at 1200℃ for a reaction-sintered mullite ceramic, which contained a level of residual silica/silicate glassy phase at the grain boundaries and junctions, has been demonstrated. The fracture strength showed a linear decrease with a decreasing loading rate ($\dot{\varepsilon}$) from 0.5 to 0.001 mm min^{-1}. This behavior fits the general relationship for steady-state creep, $\sigma \approx \log \dot{\varepsilon}$, and indicates that the decrease in fracture strength with a decreasing loading rate is dominated by a single mechanism. It was found that the energy dissipation and grain boundary sliding are two conflicting processes, both of which are associated with the plastic deformation of the residual glass phase in the transient temperature range. The former will dominate the fracture process when a high loading rate is used to fracture the material, leading to an apparent peak strength. However, the situation is reversed when the material is fractured using a sufficiently slow loading rate, which allows the time dependent grain boundary sliding to occur. Consequently, the grain boundary sliding results in slow crack growth and therefore a drop in the fracture strength.

A similar analysis for the effect of strain-rate on the mean flexural strength of glass-ceramics ($CaO\text{-}MgO\text{-}Al_2O_3\text{-}SiO_2$) has been made by C.B. Ponton [57]. The flexural strength data demonstrated that the measured flexural strength of a specimen, as represented by the mean flexural strength, increased with a growing strain-rate. The mean flexural strength of two glass-ceramics (SCR 25.76 and SCR 19.34) increased by approximately 20% for two orders of magnitude increase in strain-rate. The lower the strain-rate, the slower the

increase in the stress intensity factor of the critical flaw in a specimen as a function of increasing stress; hence the greater the degree of environmentally determined slow growth of the flaw prior to catastrophic fracture. As a consequence, the stress required to cause fractures decreases as the strain-rate decreases.

In Hanney and Morrell's study [43], the effect of the crosshead rate over two orders of magnitude was determined. The experimental results indicated that the material is susceptible to static fatigue weakening presumably because the flaws resulting in failure, being at the specimen surface in a bend test, are exposed to the environment.

2.4.2.5 Sample size

Designing structural components from brittle ceramics relies strongly on the availability of an accurate data base. However, the strength of ceramic components is usually scattered. Therefore, a considerable amount of specimens are usually needed to evaluate the mean strength and their distribution.

The choice of sample size depends on many factors including the cost and timing of testing and the degree of conservation which is acceptable, but erroneous judgments may be made and unacceptable designs pursued if the sample sizes are too small.

Ritter et al. [58] have used a Monte Carlo simulation technique to evaluate the effect of specimen number. In Lai, Lin and Tuan's study [59], an experimental investigation of the effect of specimen number on the determination of the characteristic strength and the Weibull modulus was conducted. The characteristic strength and the Weibull modulus was determined by choosing 10 or 20 or 30……100 specimens randomly from the 102 specimens. Each specimen was only chosen once. The procedures were repeated 10 times. The experimental results showed that the reliability of strength value is increased with the increase of specimen number; the reliability of the evaluation of Weibull modulus is also increased with the increase of specimen number. As the specimen number is small, the reported strength and

Weibull modulus may be significantly deviated from the true value.

In order to obtain correct values of characteristic strength and Weibull modulus, the number of specimens should be as many as possible. A sample size of 30 specimens was chosen as a compromise between obtaining narrow confidence limits and economic considerations. Improvement in confidence intervals beyond 30 specimens is on a path of diminishing returns. The MIL standard requires or recommends a minimum of 30 specimens per condition.

2.5 Summary

Flexural strength of engineering ceramics is usually measured because it is very difficult to make a test piece accurately and to grip a brittle material in the tensile test, although tensile strength is the datum required most often for designing parts.

Several test techniques for the measurement of the flexural strength of engineering ceramics have been developed. These techniques can be grouped into two methods: uniaxial flexure tests and biaxial flexure tests. There are many similarities among them, but there was little consistency in procedures or results.

Uniaxial flexure tests, such as three- or four-point beam bending tests, have long been used to measure ceramic strengths. The specimen in beam bending tests can have a circular, square, or rectangular cross section and is uniform along the complete length. The stress solution for the beam bending tests is known and well developed in the materials text books.

Biaxial flexure tests, such as ring-on-ring or 4-ball tests have obtained much attention recently since service applications of engineering ceramics generally involve multiaxial loads. They will gain considerable popularity now that the exact stress analysis is available.

The strength of ceramic components is a statistical quantity. The distribution of fracture strength of engineering ceramics is commonly described by Weibull statistics. The theory of statistical treatment of data and Weibull

distribution has been introduced.

Two material properties, the Weibull modulus and the unit strength are required for a failure probability estimate from the four-function Weibull equation. The methods for determination of these material properties have been described.

Measurement of flexural strength must be accurate if they are to be really useful and reliable. The variability in flexural strength results is often a consequence of the inherent scatter in tensile strength of brittle ceramics, but it is compounded by experimental errors in strength test methods and often inconsistency in the materials themselves.

Primary factors which have contributions to the measurement variation of flexural strengths have been presented and discussed. Among these factors, the effect of specimen size, surface finish, fixture geometry, loading rate, and sample size, which are termed selectable measurement conditions, have been a role on the relative importance and investigated in detail.

CHAPTER 3
Uniaxial Flexure Tests for Ceramics

3.1 Introduction

As mentioned earlier, the strength of engineering ceramics is often measured by the well-known flexure test method due to the high cost and difficulty of conducting direct tensile testing on engineering ceramics. The uniaxial flexure tests, i.e., beam bending tests, are the most traditional and common means to measure the uniaxial flexural strength of a brittle ceramic.

The beam in bending tests is a geometrically simple specimen, easy to manufacture and readily loaded. It can have any cross-sectional shape; however, for convenience, the section must have at least one plane of symmetry. Loading alignment is usually simple, and achieved by supporting and loading the specimen by means of smooth hard cylindrical rollers.

Galileo, in introducing the new science of the strength of materials, treated the problem of the load-carrying capacity of beams in bending [60]. Porcelain manufacturers began to use the beam bending test in the 1920s when it became evident that direct tensile testing would be experimentally difficult to conduct. In the 1950s and 1960s, the beam bending tests became a common tool of ceramic manufacturers and research laboratories. Beam bending tests were, and still are low-cost, simple, versatile methods to assess strength and quality of a material. The overwhelming majority of beam bending tests were conducted by materials scientists and processers concerned with characterization issues. They were not particularly concerned about accuracy or precision, since it was widely believed that the method was inherently accurate because of its simplicity. If there was any doubt, it was believed that the strength values could at least be used for comparative purposes [2].

A number of sources of error arose in the practical implementation of the beam bending technique whose cumulative effect can result in experimental flexural strength data totally unrepresentative of the true flexural strength.

These errors in flexure tests of beams are either due to assumptions entailed in simple beam theory, or to sources arising from external load applications. Since beam bending tests are the principal method used to determine the stress rupture and fast fracture properties of engineering ceramic materials, determination and reduction of these errors are very important.

Several test techniques for beam bending tests of engineering ceramics have been developed. There are many similarities among the techniques. Nevertheless, a myriad of test configurations arose with various specimen sizes and shapes, fixture sizes and types. There was little consistency in procedures or results. In order to obtain more consistent and accurate test results, the standardisation of flexure testing of engineering ceramics is needed.

In this Chapter, some important features of beam bending tests are briefly described. The errors associated with beam tests have been well documented and reviewed on several occasions, [e.g. 61, 62] and are only briefly summarized here. The current standardisation situation of beam bending techniques is discussed.

3.2 Beam bending tests

Determination of strength of brittle materials by three- or four-point bending test is a long-established technique and discussions may be found, for example, in Refs. 2, 41 and 42. This test method, or some variation of it refined to allow for the rocking of knife edges to accommodate warping of the specimen, is an important method because it allows measurement of strength on small bar-shaped specimens cut out of larger-shaped ceramic specimens.

It is well known that changes in specimen preparations or test procedures have had effects on the strength measurement. In order to minimize experimental error, specimen preparations and test procedures should be carefully arranged and carried out.

3.2.1 Specimen preparation

The flexure stress determined by the beam bending tests is computed based

on simple beam theory with assumptions that the material being tested is isotropic and homogeneous, and shows linear stress-strain behavior. Otherwise, the average grain size of material should be no greater than one-fiftieth of the specimen thickness.

The specimen in beam bending tests can have any cross-sectional shape; however, the rectangular section beam is by far the most common test specimen.

It is recognized that the strength of a ceramic can be dependent upon test specimen size. In general, the larger the specimen, the weaker it is likely to be. Specimen sizes and fixtures were chosen to provide a balance between practical configurations and resulting errors. Several specimen-fixture combinations were allowed since not one specimen size could meet all the needs of the engineering ceramic community. The most commonly used configuration is 3mm×4mm×45mm specimens on 20mm×40mm four-point spans or a 40mm three-point span (See Fig. 3.1).

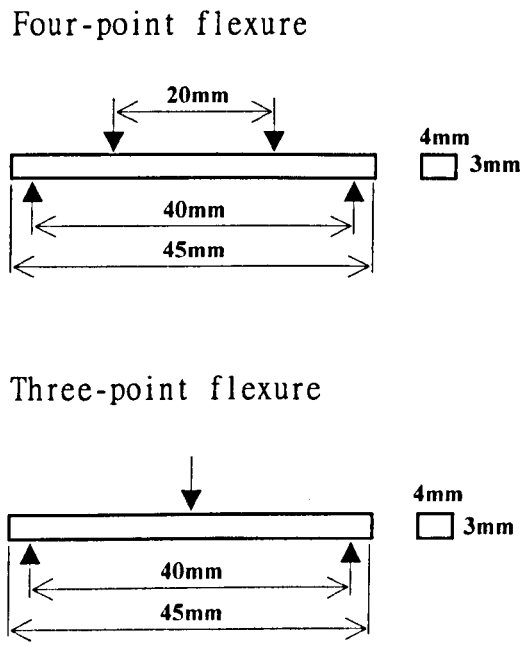


Fig. 3.1 Most common flexure configurations

Flexure specimens are especially sensitive to surface-finish preparation.

Depending upon the intended application of the flexural strength data, one of the following four specimen preparation procedures shall be used [63]:

1. As-fabricated

The flexural specimen shall simulate the surface condition of an application where no machining is to be used; for example, as-cast, sintered, or injection-molded parts. No additional machining specifications are relevant. An edge chamfer is not necessary in this instance. As-fired specimens are especially prone to twist or warping and might not meet the parallelism requirements. In this instance, a fully articulating fixture shall be used in testing.

2. Application-matched machining

The specimen shall have the same surface preparation as that given to a component. Unless the process is proprietary, the report shall be specific about the stages of material removal, wheel grits, wheel bonding, and the amount of material removed per pass.

3. Customary procedures

In the instances where a customary machining procedure has been developed that is completely satisfactory for a class of materials (that is, it induces no unwanted surface damage or residual stresses), this procedure shall be used.

4. Standard procedures

In the instances where the preceding three procedures are not appropriate, then this procedure shall apply. This procedure shall serve as minimum requirements and a more stringent procedure may be necessary.

(1) All grinding shall be done with an ample supply of appropriate filtered coolant to keep workpiece and wheel constantly flooded and particles flushed. Grinding shall be in at least two stages, ranging from coarse to fine rates of material removal. All machining shall be in the surface grinding mode, and shall be parallel to the specimen longitudinal axis (See Fig. 3.2). No Blanchard or rotary grinding shall be used.

(2) The stock-removal rate shall not exceed 0.03mm per pass to the last 0.06mm

per face. Final (and intermediate) finishing shall be performed with a diamond wheel that is between 320 and 500 grit. No less than 0.06mm per face shall be removed during the final finishing phase, and at a rate of not more than 0.002mm per pass. Remove approximately equal stock from opposite faces.

(3) Materials with low fracture toughness and a greater susceptibility to grinding damage may require finer grinding wheels at very low removal rates.

(4) The four long edges of each specimen shall be uniformly chamfered at 45°, a distance of 0.12 ± 0.03mm. They can alternatively be rounded with a radius of 0.15 ± 0.05mm (See Fig. 3.3). Edge finishing must be comparable to that applied to the specimen surfaces. In particular, the direction of machining shall be parallel to the specimen long axis. Alternatively, if a specimen can be prepared with an edge that is free of machining damage, then a chamfer is not required.

The number of specimens required by this test method has been established with the intent of determining not only reasonable confidence limits on strength distribution parameters, but also to help discern multiple-flaw population distributions. A minimum of 10 specimens shall be required for the purpose of estimating the mean of the strength. A minimum of 30 shall be necessary if estimates regarding the form of the strength distribution are to be reported (for example, a Weibull modulus).

The prepared specimens should be handled with care to avoid the introduction of damage subsequent to the machining process. The specimens should be kept separate at all times, and should be individually wrapped for transport.

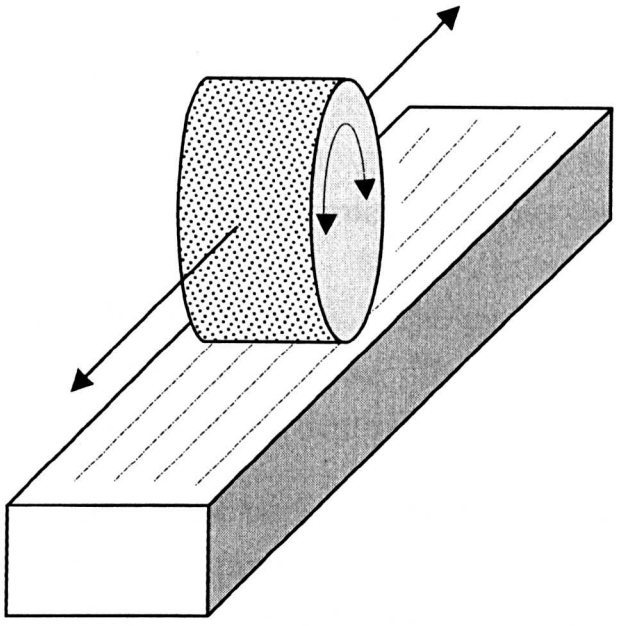


Fig. 3.2 Surface grinding parallel to the specimen longitudinal axis

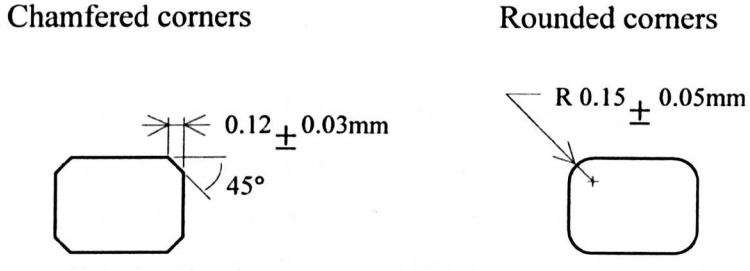


Fig. 3.3 Machined test specimens

3.2.2 Test procedure

The test apparatus shall be arranged in a suitable mechanical testing machine which shall be capable of applying a force equally to the two loading rollers in order to stress the specimen. The machine shall be capable of applying the force at a constant loading or displacement rate.

The test machine shall be equipped for recording the peak load applied to the specimen. The accuracy of the test machine shall be in accordance with EN 10002 part 2, Grade 1 (accuracy 1% of indicated load).

Cylindrical bearing edges shall be used for the support of the test specimen and for the application of load. The cylinders shall be made from hardened steel or other hard material with a hardness no less than HRC 40 or a yield strength no less than 1240 MPa. Higher strength and stiffer ceramic specimens may require harder bearings. The cylindrical bearing length shall be at least three times the specimen width. The bearing cylinder diameter shall be approximately 1.5 times the beam depth of the test specimen size.

The bearing cylinders must be free to rotate in order to relieve frictional constraints. This can be accomplished by mounting the cylinders in needle bearing assemblies. Note that the outer-support bearing roll outward and the inner-loading bearings roll inward.

The fixture shall be stiffer than the specimen, so that most of the crosshead travel is imposed onto the specimen. The testing procedure shall be in accordance with BS EN 843-1:1995.

3.2.3 Stress solution

The stress solution for the beam bending tests has been well developed [29,30]. For convenience, a rectangular cross section of width, b, and depth, d, will be considered here. From simple bending theory, the flexural strength for a rectangular test specimen can be calculated using the general flexure stress formula:

$$\sigma = My/I \qquad (3.1)$$

where M is the bending moment, y is the distance from the neutral axis to the tensile surface (See Fig. 3.4), and I is the moment of inertia. The maximum stress, σ_{max}, occurs at $y = d/2$ where the bending moment is a maximum

$$\sigma_{max} = (M_{max}/I)(d/2) \qquad (3.2)$$

Fracture occurs when the maximum stress σ_{max} equals the tensile fracture

stress of the material. For a rectangular test specimen $I = bd^3/12$ and $y = d/2$, the three-point flexure formula can be derived as

$$\sigma_f = 3PL/2bd^2 \tag{3.3}$$

and the four-point flexure formula as

$$\sigma_f = 3Pa/bd^2 \tag{3.4}$$

where σ_f=the fracture stress (N/mm^2);

P =the peak force at fracture (N);

b =beam width (mm);

d = beam depth (mm);

L =outer support span (mm);

a =the mean of the distances between centres of the inner and outer support rollers (mm).

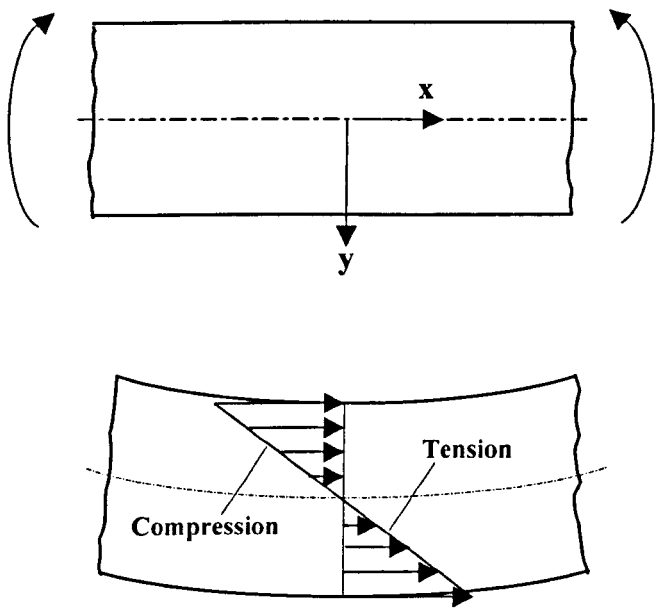


Fig. 3.4 Beam bending moment

3.3 Errors associated with beam test

Measurement of the strength of a material may be made for two purposes; to compare the relative merits of different materials, and to provide data for the adequate design of complex engineering structures. Although for comparative purposes any simple but reliable strength test will suffice, the need to provide material properties for design purposes imposes more stringent requirements.

Strength tests for engineering ceramics must have as small a testing variance as possible and allow strength parameters to be derived which are independent of the method of test. Beam bend tests, either three-point or four-point, are commonly used to obtain reliable strength data. It is important, therefore, that in a beam test, specimen shape and dimensions and the test rig design, are chosen so that deviations from the stress distribution given by elementary theory are acceptably small.

In the following sections the limitations of simple beam theory, the errors arising from the external influences and the method for minimization of experimental error are summarized.

3.3.1 Errors from simple beam theory assumptions

In the previous section the conclusion that the uniaxial stress of equations (3.3) and (3.4) determines failure in bend is based on the following simple beam theory assumptions [61].

1. Transverse planes perpendicular to the longitudinal axis of the beam remain plane after the beam is deflected.
2. The modulus of elasticity in tension is equal to the modulus of elasticity in compression. Also, the beam material is isotropic and homogeneous.
3. The maximum deflection must be small compared to the beam depth.
4. The beam must deflect normally under elastic bending stresses but not through any local collapses or twisting.
5. Stresses in the longitudinal direction are independent of lateral displacements.

Serious violation of one or more of these assumptions is responsible for the error in the apparent fracture stress.

Assumptions 1 and 2 together imply that stress and strain are proportional to the distance from the neutral axis, and the stress does not exceed the proportional limit of the material. These assumptions disregard the effect of any shearing resistance and make impossible the use of the flexure formula for curved beams of large curvature.

Assumption 1 and the above implication suggest that the bending stress is proportional to the distance from the neutral axis to the outer surface of the beam. This assumption is valid if flexure of the beam could be attained without applying local forces to the beam. However, practical flexure test systems, which utilize four-point and three-point beams, require direct contact of the fixture to the specimen to apply loads and thus moments to the specimens. At the point of contact there will be compressive stress in the beam depth direction resulting in a local variation from linearity in the bending stress.

If the beam is anisotropic, the bending stress formula is exactly the same as the elementary theory except that the application of a bending moment can produce twisting moment. Nonhomogeneity of the test material infers variation of the elastic modulus. It has been observed that in plates of hot-pressed silicon nitride, the modulus of elasticity at the surface is several percent different than that of the centre.

The validity of the assumption that the strain is proportional to the distance from the neutral axis and that stresses are independent of lateral displacements is dependent upon the ratio of the beam width to its depth (b/d). Anticlastic curvature of rectangular beams with intermediate ratios of b/d can lead to erroneous results using simple beam theory.

An error source that is internal to the beam arises because the modulus of elasticity in tension is not equal to that in compression. The occurrence of initial curvature in a rectangular beam can also result in an error. If the maximum deflection is not small compared to the beam depth, linear beam theory cannot be employed without an error. Such errors from simple beam assumptions were determined and given in Refs 61.

3.3.2 Errors from external influences

Accuracy, which is inferred in the above restrictions, is also dependent upon the manner of load application, beam geometry, loading fixtures, and surface preparation. An overview of the sources of error arising from the method of load application in bending has been given by Baratta et al. [61], Hoagland et al. [62], and Ponton [57]. In their study, it is found that the most common sources of error arising from the external influences are:

1. Friction

When determining flexural strength by simple beam theory, it is usual to assume that the supports and load points are frictionless, but in many instances they are not.

Knife-edge fixtures or fixtures with cylinders resting in "V"-grooves or slots will exert a constraint upon the specimen. During loading, the upper portion of the specimen will attempt to contract as it is stressed in compression, whereas the bottom surface will attempt to extend as it is stressed in tension. The latter extension on the bottom surface due to tensile strain is larger than the tendency to shorten due to the curvature. If these elongation are restricted by fixed-loading points, then a frictional constraint will occur. This will create a moment which will counteract the moment from the vertical forces. The stress error ($\overline{\varepsilon}$) if the friction effect is ignored is given below for the four-point loading system [61]:

$$\overline{\varepsilon} = 100 \cdot \mu / \{ (a/d) - \mu \} \qquad (3.5)$$

where μ is the coefficient of friction,

a is half the distance between the inner span and outer span for a four-point loaded beam,

d is the beam depth.

This error is systematic and all specimens of a sample will experience the same magnitude error providing that the coefficient of friction is constant. The net effect of this systematic error is to shift a Weibull cumulative distribution curve laterally [64].

This error has been previously identified by a number of authors [61,62,64 – 66]. It has been experimentally verified in a few instances at room temperature. Newnham [65] reported that the difference in failure stress using fixed knife edges as compared to roller pins was as high as 12% for silicon nitride. Quinn [42] reported an 8% error in strength of sintered alumina and verified the Weibull curve shifts by a constant factor. Swank et al. [66] investigated the experimental errors in several types of fixture designs and measured errors up to 14%.

2. Twisting

If the line loads are non-uniform, the specimen may be subject to a net torque. This might easily arise if a rectangular cross section is skewed over its length or if a pair of contact lines at one end of the specimen is not parallel to the pair at the other end [62] (See Fig. 3.5).

The error due to twisting has been estimated for plane strain and plane stress conditions by examining the maximum principal stress due to bending and torsion and comparing it to the bending stress [62]. The plane strain criterion leads to slightly higher error estimates, but the plane stress criterion is more appropriate [61].

The maximum principal stress ($\sigma_{n\,max}$) for a skewed four-point beam in bending, assuming a plane stress condition, is given by Hoagland et al. [62] as

$$\sigma_{n\,max} = \sigma_f / 2 \{1 + (1/3\, k_2)[(b^2/a^2) + 9k_2^2]^{1/2}\} \qquad (3.6)$$

where σ_f is the apparent maximum tensile bending stress, calculated using the four-point flexure formula (equation (3.4), $\sigma_f = 3pa/bd^2$), and k_2 is a numerical value associated with the torsional stress component which are dependent on the ratio of b/d.

The maximum principal stress as given by equation (3.6) can be utilized to determine the percent error for various ratios of a/b and b/d. This was accomplished and is shown in Table 10, Refs 61. Note that all errors are negative; thus for a given load the tensile stress is higher than it would be in the absence of twisting. So the fracture load and hence the apparent flexural

strength are decreased; this negative error due to twisting increases with decreasing b/d and decreasing a/b.

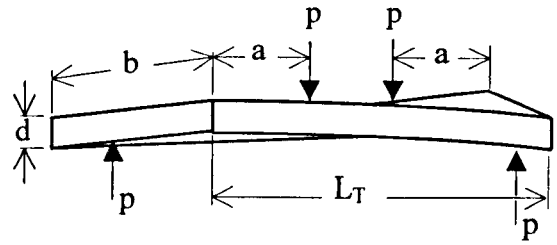


Fig. 3.5 Twisting of a four-point beam specimen

3. Wedging

In the region of contact between a knife-edge or roller and the specimen surface, the contact area is a very narrow strip and hence the stress in the contact area is extremely high; this stress concentration perturbs or distorts the general stress field in its immediate vicinity. This stress perturbation effect is known as wedging (See Fig. 3.6).

The effect of the wedging stress is to provide a substantial tensile stress contribution at the compressive side of the beam adjacent to the load points. The tensile stress is added to that already present due to beam bending at the tensile side of the beam, thereby causing a deviation from the assumed stress calculated by simple beam theory.

This problem is generally treated by S.P. Timoshenko and J.N. Goodier [67], and show that when a concentrated load is acting on a beam the resultant tensile bending stress in the x direction may be written as

$$\sigma_x = \sigma_f + (2p/bd)\,\beta_T \qquad (3.7)$$

where σ_f is the bending stress in a beam as defined by simple beam theory, 2p/bd is the wedging stress, and β_T is a numerical factor dependent on the normalized distance x'/(d/2) on either side of the applied load point.

The percent error is defined as:

$$\bar{\varepsilon} = [(\sigma_f - \sigma_x)/\sigma_x]100 \qquad (3.8)$$

substituting the equation (3.7) and (3.4) into the above equation, the percent error resulting from the wedging effect for a four-point loaded beam is given as

$$\bar{\varepsilon} = \{-\beta_T/[(3a/d) + \beta_T]\}100 \qquad (3.9)$$

It is seen that the error is dependent on β_T or the fracture location. These errors have been computed and presented in Table 11, Refs 61.

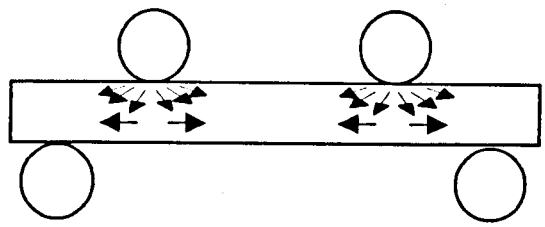


Fig. 3.6 Wedging of a four-point beam specimen

4. Edge Chamfer

Edge flaws resulting from chipping or cracking during the fabrication process are sources of low-strength failure. Rounding or beveling of the corner appears to reduce premature failure; thus the longitudinal edges of the tensile face-to-be of the specimen are frequently chamfered or rounded off to remove edge flaws which may cause premature failure of the specimens and would result in a skewed, unrepresentative average flexural strength and sample strength distribution for the material. However, the procedure reduces the moment of inertia of the cross-section and hence the true maximum tensile bending stress will be higher than the nominal maximum tensile bending stress calculated using equation (3.4).

An analysis for the error due to neglecting change in moment of inertia caused by corner radii or chamfers is given in Refs 61. The error in practice will depend on the actual profile of the "rounded" edges which may range from

a quarter-circle at one extreme to a bevel at the other. The two edge profiles may also be slightly different or have different radii; nevertheless the error incurred by rounding off the bottom edges is likely to be less than that due to premature fracture at an edge flaw.

5. Eccentric loading

When calculating bending stress by simple beam theory formula for loaded beams, it is usual to assume that the moment within the inner span is constant. However, if a loading head that can only translate is used, it is impossible to attain this idealized moment condition when a lateral displacement in the loading line relative to the perfect load location is found (see Fig. 3.7). This problem of unequal moments in four-point bending also occurs when the loads applied via the two loading knife-edge or rollers are not equal.

Lateral displacement of the loading relative to the perfect load location reduces the bending moment, which for a given load results in a lower tensile stress than there would be with no lateral shift. So the fracture load, and hence the apparent flexural strength are increased; this error increases with increasing displacement to span ratio (e／L).

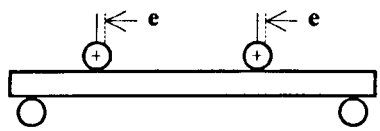


Fig. 3.7 Eccentric loading

6. Wrong spans

An additional mislocation error may exist if the inner bearing span (l) or the outer bearing span (L) are not their prescribed values, even if they are properly centered with respect to each other. This will alter the moment arm. For a four-point loaded beam, assuming the inner span is actually $l+e_s$ and the outer span is $L-e_s$, then the ratio of σ_x to σ_f is :

$$\sigma_x / \sigma_f = 1-[2\, e_s/(L-l)] \qquad (3.10)$$

where e_s is the error of the inner and outer span dimensions. A similar error

(for $e_s/L \leqq 0.01$), but of opposite sign exists if the inner span is $l - e_s$ and the outer span is $L + e_s$.

In the case of three-point loaded beams, a simple analysis shows that if the support span is actually $L - e_s$, then:

$$\sigma_x / \sigma_f = L - e_s / L \qquad (3.11)$$

If the support span is $L + e_s$, a similar error occurs but it is slightly less and of opposite sign. A comparison of equations (3.10) and (3.11) shows that the four-point configuration amplifies the span error, whereas the error in computing the stress for a three-point beam is nearly the same as the span error.

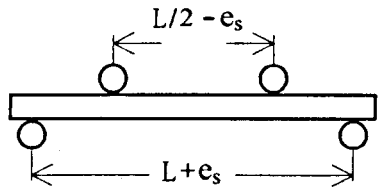


Fig. 3.8 Wrong span

7. Contact point tangency shift

Significant changes in span length can occur in both four-point and three-point loading systems if contact radii of support and load points are large compared to beam depth (See Fig. 3.9). The shift in point of tangency is a function of the contact radii, specimen thickness, and the ratio of the modulus of elasticity to the bend strength. For materials that behave elastically, such as those considered here, the change in tangency point and, thus, the error arising because of the change in moment arm from the ideal can be predicted mathematically for linear systems. This is accomplished and is presented in Refs 61.

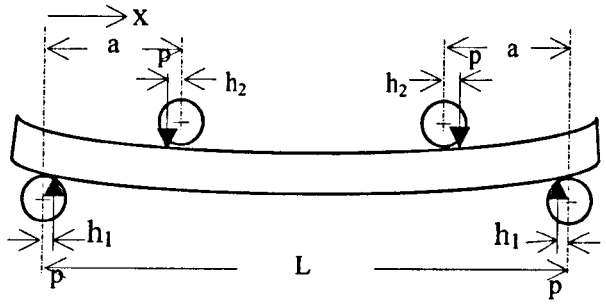


Fig. 3.9 Contact point tangency shift

8. Contact stresses

Loads on bend specimen applied through knife-edges or small-diameter rollers result in high stresses under these line loads. High compressive contact stresses can result and cause local crushing. (Also, shear stress near the locality of the load point can be several times higher than that predicted by beam theory.)

Refs 68 gives equations for determining the contact pressure between a cylinder (or roller) and a flat surface as a function of the applied load, modulus of each material, and the roller radius. If it can be assumed that the two materials are identical and that the allowable bearing pressure or contact pressure can be as high as twice the bend strength of the material, then limits on the roller radius for both loading systems will result.

9. Specimen dimension measurement

It is further evident that an error in measuring the specimen dimension can also lead to an error in stress. It is recommended that the cross section dimensions b and d be measured at the point of failure to preclude specimen taper effects. Considering the true specimen dimension to be in error by e_m, then from equations (3.3) and (3.4)

$$\sigma_x / \sigma_f = bd^2 / (b+e_m)(d+e_m)^2 \quad \text{for three-or four-point flexure} \qquad (3.12)$$

If e_m is small relative to b or d:

$$\overline{\varepsilon} = \pm[2\,(e_m / d) + (e_m / b)] \qquad (3.13)$$

Equation (3.13) shows that, if the measurement error is expressed as e_m /b or e_m /d, the error in stress is magnified. For example, if d=b, then a 1% error in specimen measurement becomes a 3% error in stress. This is what is needed throughout the test.

3.3.3 Reduction of errors in beam test

Accurate stress rupture and fast fracture data are required for the design of engineering ceramic components. Beam bending test fixtures used for testing engineering ceramic materials in flexure have inherent errors (as described in preceding section) which are reflected in a difference between the outer fiber stress calculated from simple beam formulas and the true outer fiber stress existing in the beam. Since beam bending test is the common method used to determine the stress rupture and fast fracture properties of engineering ceramic materials, reduction of these errors is very important.

Some of the more important error sources do depend upon the fixture configuration. These errors can be minimized by careful design of the bending test rig and correctly positioning the specimen with respect to the supports and the loading edge before testing it.

Hoagland et al. [62] stated that many arrangements can be devised for minimizing frictional forces. Perhaps the simplest is to design the bend fixture so that rollers are used which are completely free to roll along the specimen surface. Any inelastic processes along the contact line (either at the roller-specimen contact or the roller-fixture contact) will contribute to the rolling friction. Therefore, if the test material has a high surface hardness, very low rolling friction coefficients should be attained by the use of loading pins which also are very hard.

Swank et al. [66] used strain gages on specimens on a variety of fixture types and found that the roller supports minimize errors in calculating the true outer fibre stress of the bending test specimen. Quinn [64] also found that friction constraint causes a stress error in high temperature flexure testing and can be eliminated by the use of rolling-pin fixtures.

Baratta et al. [61] reported that the bearing friction error can be of large magnitude for either three- or four-point loading, and it is strongly recommended that the load bearings be mounted such that they are free to rotate. Twisting error, due to lack of parallelism of fixture bearings or specimen surfaces, is harder to predict, because the error is dependent upon many geometry terms as well as the specimen stiffness. Parallelism requirements are more important for four-point loading than three-point. For most geometries and materials, parallelism limits of better than 1^0 in the specimen and also the fixtures are needed to keep the error within 1 percent. The 1/3-four-point mode has somewhat less error than the 1/4-four-point mode for the cases of wrong span and contact tangency shift sources. A greater difference exists for the eccentric loading source of error. Special care should be taken to minimize wrong spans or eccentric loading error sources in four-point flexure since an error in such fixture positioning is magnified as an error of stress. Three-point loading is much less sensitive to load bearing position error sources than four-point loading. On the other hand, a three-point loaded beam is adversely affected by the presence of wedging stresses at the point of maximum stress. These wedging stresses decay rapidly with distance away from the load bearing and will have considerably less influence on four-point testing.

An experimental investigation of reduction of errors in ceramic bend tests was conducted by Hoagland et al. [62]. A bend fixture design, which incorporates tiltable pins, adjustable span position, and pins free to roll with low friction, was examined in terms of the stresses introduced by the fixture in calibration beams. A comparison of Young's moduli measured from specimens in bending with tests in uniaxial loading indicated that the deviations from the simple beam theory stress had been effectively reduced to errors of the order of 1%.

Many of the errors are independent of the test configuration. Micrometers are readily available that are accurate to within 0.0025mm, and these should be used to keep specimen dimension measurement errors to a few tenths of a percent. Corner chamfers should not be casually applied to specimens, particularly ones with small cross sections, since the error can be significant. If a specimen can be prepared with an edge that is free of machining damage,

then a chamfer is not required. Since a chamfer will double the number of edges, thus doubling the source of flaw locations, rounding is preferred. If the chamfers were not identical, or if only two chamfers are used, a further error can result due to a shift in the position of the specimen's neutral axis. Also, it is important to grind the edges by a motion parallel to, rather than perpendicular to, the specimen length. It is further indicated that finishing of the corner should be comparable in all aspects to that applied to the beam surfaces. If the corner radii or chamfer is small, the error in ignoring the change in moment of inertia will be negligible. The limiting ratio of corner radii or 45° chamfer dimension to beam depth can be determined from the error analysis due to neglecting the change in moment of inertia given in Refs 61.

3.4 Standardisation situation

The presumption that a standard test method leads to more consistent and accurate test results was validated in a four-nation, seven-laboratory round-robin exercise [2]. If a test be carefully conducted by a proper standard method, reliable and accurate results may be obtained.

Several standard methods for measuring flexural strength of engineering ceramics appeared in the early 1980s. These standards are intended to be used for material development, quality control, characterisation and design data acquisition purposes.

There are many similarities among the standards. The specimen and fixture sizes are quite comparable and many tolerances and specifications are identical. Nevertheless, there are some differences that warrant attention [63,69-76].

3.4.1 Standardisation moves forward

In 1973, a tentative unapproved set of standards was prepared by the Army Materials and Mechanics Research Center (AMMRC, then MTL) and distributed to interested and involved organizations. However, it was apparent that these tentative standards were inadequate and thus not approved.

Germany's ceramics-in-heat-engines program of the late 1970s and early 1980s prompted the German Aerospace Research Laboratory (DLR, then DFVLR), in August 1980, to issue guidelines for ambient temperature flexure testing. These guidelines were not further developed and no attempt was made to create a Deutsches Institute fur Normung (DIN) standard at the time. Nonetheless, a number of German establishments used the guidelines and they were later to have significant influence upon the U.S. Army MIL-STD [2].

The first formal test standard for engineering ceramics was JIS R1601, "Testing Method for Flexure Strength (Modulus of Rupture) of High Performance Ceramics," in December 1981. This standard was intended to be a simple, practical consensus document. It is widely used in Japan and has led to much improved consistency of results.

The U.S. Army published MIL-STD 1942 (MR), "Flexural Strength of High Performance Ceramics at Ambient Temperature," in November 1983. The MIL-STD was developed to reduce experimental error, enhance data reproducibility and consistency, and ultimately make flexure data potentially useful for design. The standard has gained widespread acceptance in the United States.

In 1989, DIN has approved a simple practical draft standard for ambient temperature four point flexure testing, DIN 51-110 part 1. France has also produced a very similar tentative standard, AFNOR B41-104.

The American Society for Testing and Materials (ASTM) created ASTM Standard C1161 in late 1990, which is based on the MIL-STD. In 1991, a Chinese National Standard CNS 12701 was established.

In 1992, a unified draft standard PrEN 843-1, "Advanced Technical Ceramics — Mechanical Properties of Monolithic Ceramics at Room Temperature—part 1: Determination of Flexural Strength," has been prepared by the European Committee for Standardisation (CEN).

Two standards for flexure testing at elevated temperatures, JIS R1604-1987 and ASTM C1211-92, have been developed which are clones of the respective room temperature standards.

The MIL-STD 1942 (MR) was updated in 1990, which with some minor changes to make it more consistent with the other standards, and to make it more readable. Both the JIS R1601-1981 and JIS R1604-1987 were updated in 1995. The ASTM C1161-90 was also revised in 1994 and 1996. The draft standard PrEN 843-1:1992 has been formally adopted by CEN as European Standard EN 843-1:1995 in July 1995.

An international draft standard ISO/DIS 14704, "Fine Ceramics—Test Method for Flexural Strength of Monolithic Ceramics at Room Temperature," has been developed by ISO/TC 206. A new work item proposal for test method at elevated temperatures has been approved at the third plenary meeting in July 1996 and is currently being prepared by working groups WG7 of ISO/TC 206.

3.4.2 Comparison of existing standards

A comparison of flexure testing standards was shown in Table 3.1. It is recognized that flexural strength of a group of test specimens is influenced by several parameters associated with the test procedure. Such factors include the loading rate, test environment, specimen size, specimen preparation, and test fixtures.

Test fixtures and specimen sizes should be chosen to provide a balance between practical configurations and resulting errors. The three-point loaded beam system is preferred when investigating material or process development, because of smaller specimen size, or when attempting to pinpoint fracture origin location. On the other hand, the four-point loaded beam is preferred when determination of strength for design purposes is desired, because the centre span is uniaxially stressed. Each of these systems is suited for a particular application and each has different advantages and disadvantages. In MIL-STD, ASTM, JIS, AFNOR, and EN, the specimen can be tested in either three- or four-point fixture. However, the specimens of DIN 51-110 were to be tested only in four-point fixture. In MIL-STD, the four-point test is the preferred mode of testing since the larger amount of material that experiences the maximum stress in four-point loading.

Some of the more important error sources do depend upon the fixture configuration. The 1/3-four-point mode has somewhat less error than the 1/4-four-point mode for the cases of wrong span and contact tangency shift sources. In JIS R1601 and CNS 12701, the 1/3-four-point mode is the standard.

The frictional constraints can cause experimental errors in the order of 10% to 20%. It is strongly recommended that the load pins be mounted such that they are free to rotate in order to eliminate undesirable friction error. If the specimen is warped, twisted or cannot meet the parallelism requirements, then a fully articulating fixture may be required to minimize errors due to non-uniform load application along the bearing edges. The ASTM, MIL-STD, DIN, AFNOR and EN standards require the load rollers to be free to rotate to eliminate friction errors that can be present with the JIS R1601, even with the required polished load points.

It is recognized that the strength of a ceramic can be dependent upon test specimen size. In general, the larger the specimen, the weaker it is likely to be. Such size influence can be analyzed via statistical theories of strength. In the interests of permitting greater compatibility of data, specific specimen and fixture sizes will be required by the standard. In MIL-STD, several specimen-fixture combinations were allowed since it was believed that no one specimen size would meet all the needs of the engineering ceramics' community. Nevertheless, configuration A is not preferred since it is to be used only when a larger configuration is not obtainable. In ASTM, the mid-sized B test configuration, which has 3mm×4mm×45mm specimens on 20mm×40mm four-point spans or a 40mm three-point span, has proved to be the most popular, and commercial fixtures for both room- and high-temperature testing are available. In JIS R1601, one specimen size, 3mm × 4mm × 35mm was prescribed, which could be tested in either four-point fixture with 10mm× 30mm spans or three-point fixture with 30mm span. In DIN 51-110 part 1, a 3mm×4mm×45mm specimen was the standard and was to be tested only in four-point fixture with 20mm×40mm spans. In AFNOR B41-104 and EN 843-1, the specimen sizes and spans are similar to MIL-STD test configuration A and B.

Test specimen surface preparation can have a pronounced effect upon flexural strength due to the introduction of machining flaws which can be strength limiting. Surface preparation can also lead to surface residual stresses. Universal optimum or standardised methods of surface preparation do not exist. Nevertheless, some minimum requirements will be specified in the standard. The MIL-STD, ASTM, DIN, AFNOR and EN standards make provision for as-fired, twisted, or warped specimens by the use of articulated fixtures, whereas the JIS standard can be used only with well-aligned fixtures and almost perfect specimens. There are differences in the preparation procedures if machining is required. The MIL-STD, ASTM, DIN and EN standards are similar and give a prescribed two- or three-step, progressively finer process. The AFNOR standard prescribes a two-step polishing procedure, although alternative grinding procedures are permitted. However, the JIS standard is quite different in that it prescribes a final surface finish only. It cannot rule out the possibility that machining damage could exist under the surface.

The choice of sample size depends on many factor including the cost and timing of testing and the degree of conservation which is acceptable, but erroneous judgements may be made and unacceptable designs pursued if the sample sizes are too small. Statistical analysis show that wide variances in mean strengths and Weibull parameters are normal for samples with as few as 10 specimens. The number of specimens required for the flexure testing has been specified. In JIS and AFNOR, a minimum of 10 specimens shall be required. In DIN 51-110 standard, the minimum number of specimens is 15, but 30 are preferred. In MIL-STD, ASTM, and EN, a minimum of 10 specimens shall be required for the purpose of estimating the mean of flexural strength, a minimum of 30 shall be necessary if estimates regarding the form of the strength distribution are to be reported (fox example, a Weibull modulus).

Corner flaws resulting from chipping or cracking during the grinding operation are sources of low-strength failure. Rounding or beveling of the corner appears to reduce premature failure. If a specimen can be prepared with an edge that is free of machining damage, then a chamfer is not required. Since a chamfer will double the number of edges, thus doubling the source of flaw locations, rounding is preferred. If the corner radii of chamfer is small,

the error in ignoring the change in moment of inertia will be negligible. The limiting ratio of corner radii or 45° chamfer dimension to beam depth can be determined from the error analysis due to neglecting the change in moment of inertia. The chamfer sizes in the MIL-STD, JIS R1601, DIN 51-110 and AFNOR B41-104 are liberal (up to 0.3mm), and a 4% error in stress is possible. However, in ASTM C1161 and EN 843-1, the chamfer size have been reduced relative to those allowed in MIL-STD 1942 (MR). In those two standards, the four edges of each specimen shall be uniformly chamfered at 45°, a distance of 0.12 ± 0.03mm. They can alternatively be rounded with a radius of 0.15 ± 0.05mm.

The rate of loading can have an effect on flexure strength as the result of stress corrosion mechanisms, particularly at low strain rates. In general, the slower the speed of loading, the greater the opportunity for stress corrosion phenomena to weaken the specimen. Thus, fast loading speeds are usually used in strength tests. Timed failure rates for typical ceramics will range from 3 to 30 seconds. Selection of the crosshead rate may have to be determined by experiment, depending on the elastic compliance of the test machine, the stiffness of the test jig and the elastic properties of the test specimens. A crosshead rate of typically 0.5mm/min is a convenient starting point for most testing machines in cases where the expected strength of the material is 200-400MN/m^2. For material which is much weaker or much stronger than this, the loading rate may have to be respectively decreased or increased by an appropriate factor. In MIL-STD, ASTM, JIS, AFNOR and EN, a loading rate of 0.5mm/min was the standard. In DIN 51-110, the loading rate is controlled such that fracture is obtained in a time period of 5-10s.

	United State	Japan	Germany	France	CEN	Taiwan
Title (year)	MIL-STD 1942 (1990) ASTM C1161 (1994)	JIS R 1601 (1995)	DIN 51-110 Part 1 (1989, draft)	AFNOR B41-104	EN 843-1(1995)	CNS 12701 (1991)
Geometry	Three or 1/4-four point	Three or 1/3-four point	1/4-four point	Three or 1/4-four point	Three or 1/4-four point	Three or 1/3-four point
Span (four point)	10 mm x 20 mm 20 mm x 40 mm 40 mm x 80 mm 0.75 in x 15 in	10 mm x 30 mm	20 mm x 40 mm	10 mm x 20 mm 20 mm x 40 mm	10 mm x 20 mm 20 mm x 40 mm	10 mm x 30 mm
Fixture	Semiarticulating or fully articulating	Fixed	Fully articulating	Fully articulating	Fully articulating	Fixed
Load bearing	Rotating	Fixed	Rotating	Rotating	Rotating	Fixed
Specimen sizes	1.5 mm x 2 mm x 25 mm 3 mm x 4 mm x 45 mm 6 mm x 8 mm x 90 mm 0.13 in x 0.25 in x 2 in	3 mm x 4 mm x 35 mm	3 mm x 4 mm x 35 mm	2 mm x 4 mm x 25 mm 3 mm x 4 mm x 45 mm	1.5 mm x 2 mm x 25 mm 3 mm x 4 mm x 45 mm	3 mm x 4 mm x 36 mm
Specimen preparation	(a) As-fired (b) Application matched (c) Three steps prescribed (wheels, grits, rates, etc) Others	Surface roughness less than 0.8 s	(a) As-fired (b) Three steps prescribed (wheels, grits, rates, etc)	(a) Polish, deeply (tensile face) (b) Grinding optional (c) others	(a) As-fired (b) Machined (c) Finishing by grinding (d) Finishing by lapping/polishing	Surface roughness less than 0.8 s
Specimen chamfers	Up to 0.15 mm (ASTM) Up to 0.3 mm (MIL-STD)	Up to 0.3 mm	Up to 0.3 mm	Up to 0.3 mm	Up to 0.15 mm	Unspecified
Specimen size (minimum)	10 for mean 30 for Weibull analysis	10	15, preferably 30	10	10 30 for Weibull analysis	10
Loading rate	0.2 mm/min (for A size) 0.5 mm/min (for B size)	0.5 mm/min	5 – 10 s	0.2 mm/min 0.5 mm/min (or higher)	0.5 mm/min	0.5 mm/min

Table 3.1 A comparison of flexure testing standard

3.5 Summary

Accurate stress rupture and fast fracture data are required for the design of engineering ceramic components. Generally, this type of data is obtained by testing in flexure on modulus of rupture fixtures. These fixtures stress a rectangular beam in three- or four-point bending. The stress solution for the beam bending tests has been well developed.

Several types of errors occur in the beam bending tests, which cause the true stress in the outer fibre of the beam to be different from the stress calculated from simple beam theory formulae. These errors are either due to simple beam theory assumptions, or to sources arising from external influences. The limitations of simple beam theory, the most common sources of error arising from external load applications and the method for minimization of these errors have been described.

Many standards for beam bending tests of engineering ceramics have been established. These standards may be used for material development, quality control, characterization, and design data generation purposes. Standard test methods now available will hopefully unify test practice. Their use is strongly encouraged.

The standardisation processes differ considerably in the different countries, and therefore certain aspects of the standard can vary significantly.

From the comparison of existing flexure testing standards, we find that the ASTM C1161 and MIL-STD 1942 have more options in the possible testing configuration and the JIS R1601 standard is less stringent in some technical details but is simple to use. The test methods of DIN 51-110 part 1, AFNOR B41-104 and EN 843-1 are similar to MIL-STD B test configuration. With the exception of the potential chamfer error and surface preparation effects, it is expected that the test results of ASTM C1161, MIL-STD 1942 B test configuration, DIN 51-110 part 1, AFNOR B41-104 and EN 843-1 will be completely compatible.

CHAPTER 4
Biaxial Flexure Tests for Ceramics

4.1 Introduction

Measuring the strength of engineering ceramics by the uniaxial flexure tests is a long-established technique. This test method for the measurement of flexure strength of high performance ceramics has been standardised by JIS, ASTM, DIN and AFNOR etc. This test method is an important method because it allows the measurement of strength on small bar-shaped specimens cut out of larger-shaped ceramic specimens. The measured strength depends, however, upon both the condition of the surface in tension and the condition of the edges in tension. It is difficult to separate edge and surface effects, so a second method which would not be dependent upon edge condition is needed.

The relative ease of manufacture of rectangular beams makes the beam particular attractive as a test specimen. However, for many ceramic materials the production of thin circular plates as test specimens may be even easier by die-pressing the ceramic powder to the form needed for test and possibly more advantageous, though their widespread use is restricted by the test methods available with well-established stress solutions.

In addition, the loading under service conditions is seldom of pure bending, compression or torsion, respectively. Consequently, the conventional uniaxial beam bending test is often of limited value for the design engineer. Thus, the biaxial flexure test on discs with biaxial stresses is advisable.

Watchman [33] has examined many possible ways for the biaxial flexure tests. This test method involves supporting a plate on three or more points near its periphery and equidistant from its centre and loading a more central portion. The area of maximum tensile stress thus falls at the centre of the lower face of the plate and the strength should be independent of the condition of the edges of the plate. A number of variations of this technique exist. Five such test methods are the ring-loaded ring-supported (ring-on-ring test), the ball-loaded

disc supported by three equispaced balls (4-Ball test), the ball-loaded ring-supported (ball-on-ring test), the piston-loaded ring-supported (piston-on-ring test), and the piston-loaded disc supported by three equispaced balls (piston-on-3-ball test).

The piston-on-ring test has been applied by Wilshow [34] to measure the strength of polycrystalline alumina discs. He used a ball having a mechanical flat to apply the load. The piston loading is effectively a ring-load, as only line contact is obtained. The piston-on–3-ball test has been accurately analysed for small deflections (less than the specimen thickness). This method has an advantage in that support of the specimen on three balls allows the use of a slightly warped specimen. Thus no surface grinding or polishing is required, in contrast to the ring supported techniques [33]. This method has been adopted as an ASTM F394-78 (Reapproved 1996) standard for biaxial flexure strength testing of ceramic substrates. However, both the piston-on-ring test and the piston-on-3-ball test have the disadvantage that the load distribution under the piston is uncertain and difficult to model. The fact that the load is not uniformly distributed is evidenced by two experimental results [77]. The maximum strain measured at the centre of the specimen showed significant scatter. Second, all measured strains were greater than the predictions of the Kirstein and Woolley solution [78], which assumes uniform loading. These results suggest that, experimentally, it is not possible to produce uniform loading under a piston.

An improved test fixture for biaxial-tension strength testing of ceramics featuring uniform hydraulic pressure loading of discs was developed and qualified by D.K. Shetty et al. in 1983 [37]. In their work, biaxial data were obtained for an alumina ceramic, along with comparable uniaxial data from three- and four-point flexure tests.

In this chapter, the theoretical analysis and experimental investigation of three major techniques of the biaxial flexure tests for ceramics, ie. ring-on-ring, ball-on-ring, and 4-Ball test are described. The effects of varying the test parameters are also discussed.

4.2 The ring-on-ring test

4.2.1 Introduction

The ring-on-ring test involves supporting a circular plate on a ring and loading with a small concentric ring. For most ceramics, strength depends on the effective stressed area or volume because of the statistical distribution of strength-controlling flaws. The ring-on-ring loading fixture was constructed to provide a biaxial-tension-strength test in which the effective stressed area or volume of the specimen is comparable to the conventional uniaxial-flexure tests, such as four-point beam bend tests. A theoretical analysis of the ring-on-ring loading disc test has been conducted by Fessler and Fricker [79]. Quinn and Wirth used this test to determine the equi-biaxial strength of hot-pressed silicon nitride at high temperature [80].

The ring-on-ring test is gaining considerable popularity as an exact analytical stress solution is available for it. In the following, a theoretical stress analysis for the ring-loaded, ring-supported circular discs is demonstrated. The test specimen and testing jig designs for a room-temperature test, which could also be used for high-temperature testing, are described. The Weibull modulus and the unit strength of alumina ceramics subjected to a serious of ring-on-ring test are presented. The effects of the radius of support ring and load ring on fracture strength are also discussed.

4.2.2 Stress analysis

The ring-loaded ring-supported circular plate has an analytical stress solution [81]. By superposition of two cases it is possible to allow for material overhanging the support. The derivation of the analytical solution for the deflection, bending moment and stresses for this plate are given in Appendix 3 [12].

Consider a thin circular plate, simply supported and loaded as shown in Fig. 4.1:

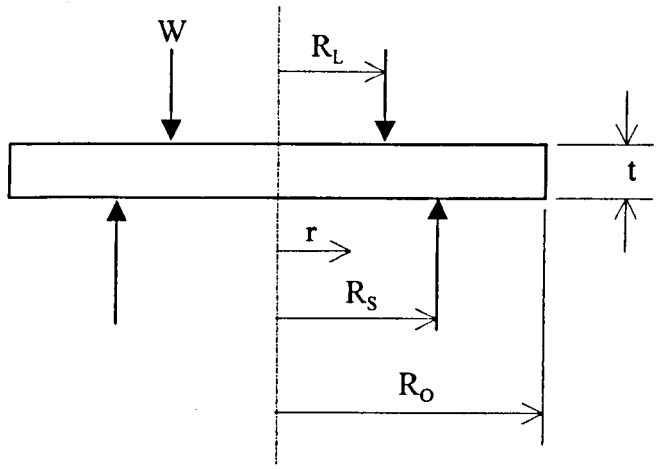


Fig. 4.1 The ring-loaded ring-supported disc in bending

it follows from Appendix 3 that within the central portion of the ring-loaded ring-supported disc specimen, i.e. for $r < R_L$, there is a uniform equi-biaxial stress state which varies linearly from tension on the lower supported surface through zero on the mid-surface to compression on the upper surface. The lower surface tensile stress, σ, is related to the applied load W and the specimen dimensions by the expression.

$$\sigma = (3W/2\pi t^2)[(1+\nu)\ln(R_S/R_L) + (1-\nu)(R_S^2 - R_L^2)/2R_O^2] \qquad (4.1)$$

where W : applied load
t : thickness of the disc plate
ν : Poisson's ratio of the disc plate
R_S : radius of the support ring
R_L : radius of the load ring
R_O : radius of the disc plate

Hence, the tensile fracture strength σ_f is readily obtained from the fracture load W_f using equation (4.1) if the Poisson's ratio is known or a value assumed for it.

Vitman and Pukh [82] verified equation (4.1) in part by comparing derived stresses with those calculated from strain gage measurement on glass specimens. Good agreement was obtained. However, strain was measured only at the centre of the disc. In W.H. Duckworth et al.'s study of ring-on-ring testing of lenses [83], a stress magnification was noted in the annular region directly below the loading ring. To examine this finding, strain measurement were made on a disc specimen of 4340 steel having dimensions 51mm in diameter and 2.54mm in thickness by D.K. Shetty et al. [77]. They found the agreement between theory and experiment is within 10% at all locations, except under the loading ring. At this location, the tangential stress component is approximately 21% greater than the predicted value. A consequence of this stress concentration is that the area and volume of the specimen subjected to maximum tension in the ring-on-ring test are not as large as simple plate theory suggests.

In Fessler and Fricker's study [79], the theoretical thin-plate analysis of axi-symmetrically loaded flat circular plates is extended to a thick plate solution by including shear between the load and support rings. The effect of friction at the contacts is also included and shown to be important. It was found that friction at the loading and support rings reduces the hoop and radial stresses inside the loading ring, and the shear stresses between the loading and support rings do not increase the maximum stresses and are unimportant.

Sivill [12] also carried out a stress analysis, using the finite element method, for a thick disc loaded in such a manner. The structure was represented by 105 8-noded isoparametric finite elements in an orthogonal mesh with 5 elements through the thickness and 21 along a radius. An additional facility was written into the program to calculate stress-volume and stress-area integrals directly.

4.2.3 The test specimen

An important characteristic of engineering ceramics is that their fracture is not preceded by any significant plastic deformation. Hence, any locally induced high stresses, caused by misalignment of the specimen in the loading jig or due to contact effects of the loading attachments themselves, cannot be relieved by plastic flaw and result in premature failure of the specimen.

Two essential features of brittle test specimens are [13]:

1. Geometry and loading are such that at the fracture section of the specimen there is a known or calculable stress state such that the fracture strength can be calculated from the fracture load.
2. The specimens are geometrically simple and that the minimum is required by way of loading attachments so as to eliminate the risk of premature failure due to loading misalignment or contact effects.

The fracture specimens, used in this study of the ring-on-ring test, were prepared by die-pressing an alumina powder (99.8% Al_2O_3, US-3061C, Showa Denko, Japan) onto a disc plate. In order to obtain the better quality of specimens, the pressing pressure employed was 196 MPa. The green compacts were sintered at 1600°C for 2 hours. The fired specimens were machined with #600 resin-bonded diamond wheel at a cutting depth of 1 μm/pass. Equal stock were removed from both the test surface, tension (support) and compression (loaded). The final dimensions of the disc were about 43mm in diameter and 2.2mm thick.

It was required that all specimens be manufactured from the same powder lot and was pressed at the same condition so as to eliminate any possible batch-to-batch strength variability.

4.2.4 Testing jig designs

The testing jig used in the work is shown in Fig. 4.2. The jig was designed to have a 40mm or 30mm outer ring diameter and a 20mm or 10mm inner ring diameter. The rings were made from die steel (hardened and tempered) and top-surface of the ring is radiused to 5mm. A high carbon chrome alloy steel ball with a 10mm diameter was used to apply the load centrally to the loading ring. The load and support act through 2mm radius toroids to minimize friction. The most important consideration in the design was that there should be no eccentricity of loading and the self-aligning of the planes must be achieved.

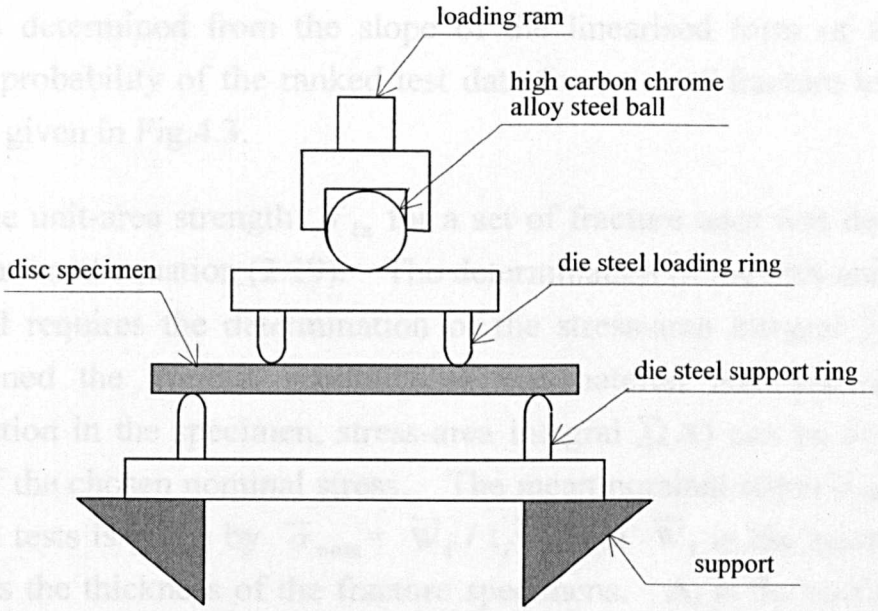


Fig. 4.2 Schematic of the ring-on-ring test jig

4.2.5 Determination of material properties

The strength of the specimens was determined by ring-on-ring testing at ambient room-temperature conditions with a new produced desktop testing machine (Engineering system (Nottm), model CK10) shown as Plate 4.1.

For economic consideration, a set of only ten pieces of disc specimen was tested. Each disc specimen was centralized in the jig relative to the support. The disc specimens were all loaded to fracture on the same CK10 testing machine with a 40mm outer ring diameter and a 10mm inner ring diameter at a loading rate of 0.5 mm/min.

All stresses were computed from the mentioned equation (4.1). No data were to be discarded. The values of Poisson's ratio, assumed in equation (4.1), was 0.26 for the disc plate. The individual fracture loads and fracture strength, σ_f, for disc in bending are given in Table 4.1. The mean strength, $\overline{\sigma}_f$ for a set of tests of sample size 10 are tabulated in Table 4.2 together with the associated standard deviations and coefficient of variation (C.O.V) of mean strength.

The material properties, m and $\overline{\sigma}_{fa}$, were determined from each set of fracture tests in the manner described in Section 2.3.5. The Weibull modulus, m, was determined from the slope of the linearised form of the cumulative failure probability of the ranked test data for a set of fracture tests. A set of plots is given in Fig.4.3.

The unit-area strength $\overline{\sigma}_{fa}$ for a set of fracture tests was determined from the mentioned equation (2.29). The determination of the unit-area strength of a material requires the determination of the stress-area integral $\Sigma(A)$. Having determined the Weibull modulus of the material and knowing the stress distribution in the specimen, stress-area integral $\Sigma(A)$ can be evaluated on the basis of the chosen nominal stress. The mean nominal stress $\overline{\sigma}_{nom}$ for a set of fracture tests is given by $\overline{\sigma}_{nom} = \overline{W}_f / t_f^2$ where $\overline{W}_f$ is the mean fracture load and t_f is the thickness of the fracture specimens. A_f is the surface area of the fracture specimens including the overhang. The calculated mean fracture load, Weibull modulus and unit-area strength for a set of fracture test is given in Table 4.3.

Specimen No.	Specimen dia. (mm)	Specimen thickness (mm)	Fracture load (N)	Fracture strength (MPa)
H1	43.35	2.17	1515	314
H2	43.38	2.25	1588	306
H3	43.58	2.23	1865	365
H4	43.68	2.25	1613	310
H5	43.19	2.20	1491	301
H6	43.56	2.17	1725	357
H7	43.50	2.17	1539	318
H8	43.62	2.16	1595	333
H9	43.35	2.26	1915	366
H10	42.53	2.16	1352	284

Table 4.1 The fracture loads and fracture strength for discs in ring-on-ring testing

Test model	Mean strength (MPa)	Standard deviation (MPa)	Coefficient of variation (%)
ring-on-ring test outer ring dia.=40mm inner ring dia.=10mm loading rate =0.5mm/min	325	29	8.9

Table 4.2 The mean strength for a set of ring-on-ring tests

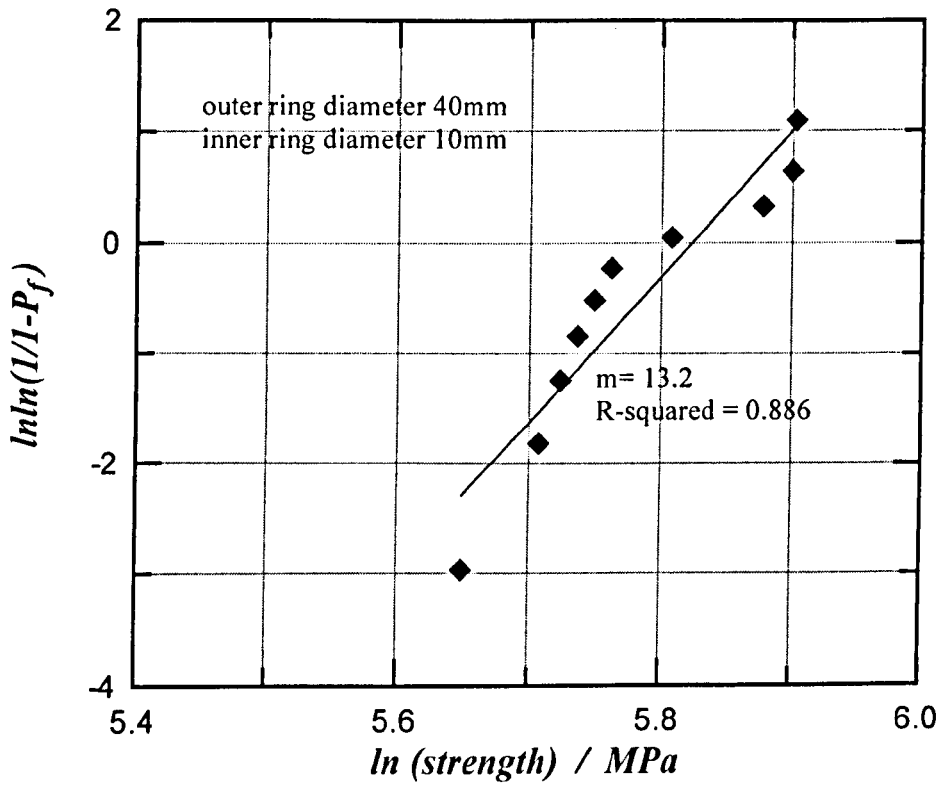


Fig. 4.3 The Weibull curve for the ring-on-ring test

Test model	Mean fracture load (N)	Weibull modulus	Unit-area strength (MPa)
ring-on-ring test outer ring dia.=40 mm inner ring dia.=10 mm loading rate=0.5 mm/min	1620	13.2	333.9

Table 4.3 The unit-area strength for a set of ring-on-ring tests

4.2.6 The effects of the radius of support ring and load ring on fracture strength

The measured strength will vary significantly depending on the fixture geometry as described in Section 2.4.2.3. It was desired to investigate the effects of the radius of support ring and load ring on fracture strength by varying the fixture size of test jig.

Thirty discs were tested to evaluate the influence of fixture sizes. The ring-on-ring tests were performed using CK10 test machine on these disc specimens at a loading rate of 0.5 mm/min with various fixture sizes of model No.1 (outer ring diameter 40mm, inner ring diameter 10mm), No.2 (outer ring diameter 30mm, inner ring diameter 10mm) and No.3 (outer ring diameter 40mm, inner ring diameter 20mm) respectively. Ten pieces of specimen were tested for each fixture size.

The fracture results were analysed as before (Section 4.2.5). The individual fracture results are given in Appendix 4. The estimated parameters of this investigation are given in Table 4.4 and shown in Fig. 4.4.

It is found that the fixture size of the ring-on-ring test jig did strongly influence the fracture strength. For the same loading rate, the larger the outer ring diameter to inner ring diameter ratio, the greater the mean fracture strength obtained. The ring-on-ring test with outer ring diameter of 40mm and inner ring diameter of 10mm showed the smallest value of coefficient of variation.

The dependence of fracture strength on the fixture size was associated with variations in the stress distribution in the test specimen and the flaw size distribution of the material. The area and volume under peak tensile stress or near peak tensile stress is greater for the ring-on-ring test jig with smaller the outer ring diameter to inner ring diameter ratio, and thus the probability of a larger flaw being exposed to high stress is increased. As a result, the fracture strength measured in the ring-on-ring test jig with larger outer ring diameter to inner ring diameter ratio is greater than that measured in smaller ring diameter ratio. The smallest value of coefficient of variation obtained from the test with the outer ring diameter of 40 mm and inner ring diameter of 10 mm was attributed to the narrowest flaw size distribution under ring-on-ring test.

Model No.	Mean strength (MPa)	Standard deviation (MPa)	Coefficient of variation (%)	Weibull modulus
1 outer ring dia.= 40mm inner ring dia.= 10mm	325	29	8.9	13.2
2 outer ring dia.=30 mm inner ring dia.=10 mm	296	47	15.9	7.4
3 outer ring dia.=40 mm inner ring dia.=20 mm	229	24	10.5	10.9

Table 4.4 The fixture size effect of fracture strength in ring-on-ring test

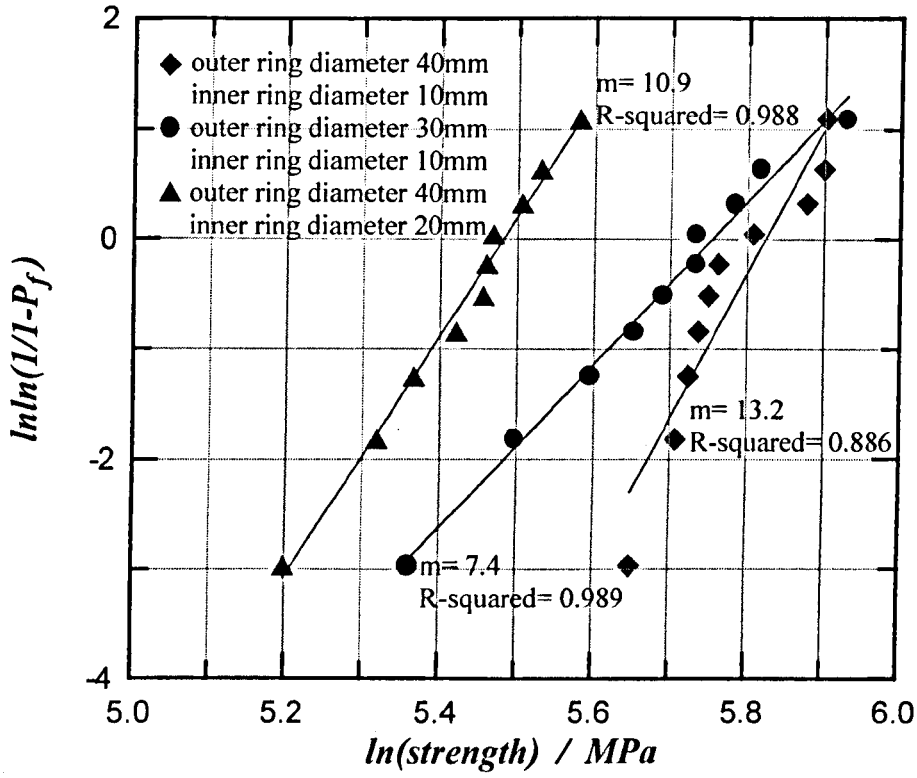


Fig.4.4. The Weibull curves for the ring-on-ring test using various fixture sizes

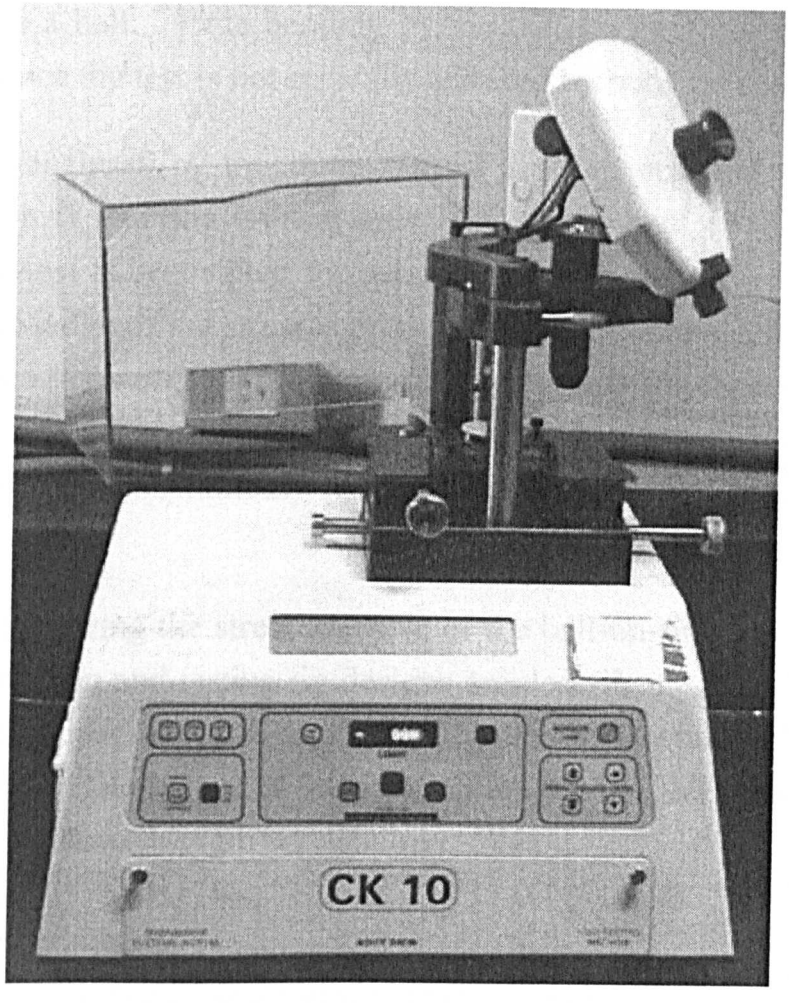


Plate 4.1 A desktop testing machine CK 10

4.3 The ball-on-ring test

4.3.1 Introduction

The ball-on-ring test involves supporting a disc plate on a ring and centrally loading with a ball. Plate bending by the ball-on-ring method is an attractive technique since the test is not critically affected by poor specimen tolerance.

In the analyses of the three biaxial loading schemes, i.e. ball-on-ring, piston-on-3-ball, and ring-on-ring tests, D.K. Shetty et al. [77] suggested that the ball-on-ring test is best suited for ceramic strength testing. Its advantages are precise knowledge of the stresses produced in the specimen, simple test fixtures and specimen geometry, and minimum requirements for alignment. The stress distribution can be accurately evaluated by finite element analysis of a point-loaded disc. An axisymmetric two-dimensional model is adequate to calculate the stress.

In the following the stress analysis of the ball-on-ring test is demonstrated. The test specimen and testing jig designs are described. The Weibull modulus and unit strength of alumina ceramics subjected to a series of ball-on-ring tests are presented. The effects of load ball diameter and the nature of the support and load on fracture strength are also investigated.

4.3.2 Stress analysis

A.F. Kirstein and R.M. Woolley [78] presented a special application of a more general solution developed by Bassali [84] for symmetric bending of a thin circular elastic plate supported at several points equally spaced along a concentric support circle and subjected to a transverse load which is symmetrically distributed over a concentric circle area. Equations for deflection, bending moments and stresses are derived in Appendix 5, taking into account both the distributed load and the overhanging material.

For a thin circular disc, supported and uniform concentric loaded as shown in Fig. 4.5:

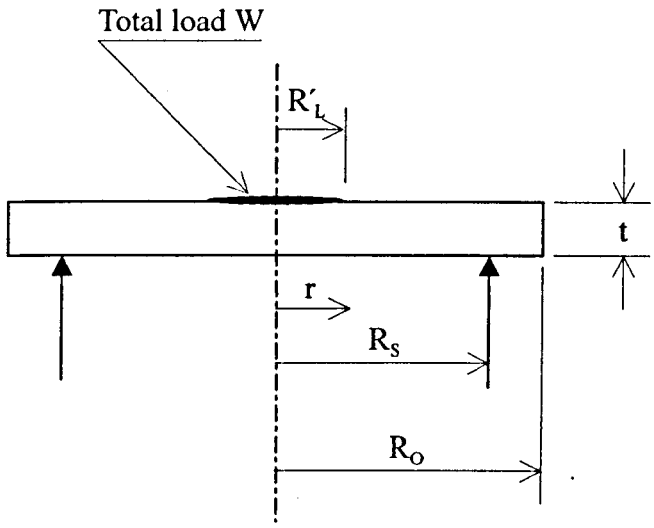


Fig. 4.5 The ball-loaded ring-supported disc in bending

the maximum radial and tangential stresses at the centre are equal and given by [78]

$$\sigma_{max} = \frac{3W(1+\nu)}{4\pi t^2}\{1 + 2\ln\frac{R_S}{R'_L} + (\frac{1-\nu}{1+\nu})[1 - \frac{R'^2_L}{2R_S{}^2}]\frac{R_S{}^2}{R_O{}^2}\} \qquad (4.2)$$

where W: applied load

t : thickness of the disc plate

ν : Poisson's ratio of the disc plate

R_S: radius of the support ring

R_O: radius of the disc plate

R'_L: contact radius of the ball

Details of the ball contact were determined from a formulas given by Roark [85]. The radius of contact of the ball (R'_L) is

$$R'_L = 0.721[Wd((1-\nu_b^2)/E_b + (1-\nu^2)/E)]^{1/3} \qquad (4.3)$$

where d is the ball diameter, E_b and E are the Young's moduli of the ball and plate respectively, ν_b and ν are the Poisson's ratio of the ball and plate respectively.

The tensile fracture strength, σ_f, (i.e. the maximum stress on the lower surface of the disc at fracture) can be estimated from equations (4.2) and (4.3) if the fracture load, W_f, and the Young's moduli and Poisson's ratio of the ball and disc materials are known or values assumed for them.

To examine the applicability of the stress solution, a stress analysis which included experimental evaluations from strain measurements and finite element procedures was conducted by D.K. Shetty et al. [77]. Strains were measured on a disc specimen of 4340 steel (Young's moduli E= 203.4 GPa and ν= 0.3) having dimensions R= 25.4mm and t = 2.54mm. The ball-bearing race support radius was 19mm. A total of seven strain gauges were used for measuring radial and tangential strains at four radial distances. Stresses calculated from the measured strains are compared with analytical solutions of equations (4.2) and (4.3). A two-dimensional axi-symmetric model was generated to conduct the finite analysis of the disc specimen in the ball-on-ring test. The finite element mesh is more refined in the load-support regions to allow a more accurate representation of the high stress gradients at these sites. Linear elastic behavior was assumed. A Hertzian-type load distribution was applied. A computer subroutine translates this distribution into a point force distribution consistent with the applied load. The results agree with the experimental values when point loading is assumed. It is also found that the Kirstein and Woolley solution (equation (4.2)) was fitted to the experimentally measured maximum stress by setting contact radius R'_L equal to 0.31 t.

Sivill [12] also carried out a finite element analysis for the stress determination of the ball-on-ring test. The structure was represented by 105 8-node isoparametric finite elements with 5 elements through the thickness and 21 along a radius. Each element was rectangular and a high concentration of elements was used at the loading point. The three principal stresses were computed on a regular grid at 16 positions within each element, making a total of 1680 points of known stress. An additional facility was written into the program to calculate the stress-volume and stress-area integrals of the plates directly.

A complete photoelastic stress analysis was taken for ball-on-ring test by C.G. Karroum [13]. By means of the well-established photoelastic stress-

freezing techniques, the determination of stress can be carried out from the reading of isochromatic fringe patterns through a polariscope.

4.3.3 The test specimen

The specimens were prepared by die-pressing an alumina powder (99.8% Al_2O_3, US-3061C, Showa Denko, Japan) onto a disc plate. The preparation and surface finish of disc specimens were the same as in the ring-on-ring testing. The pressing pressure employed was 196 MPa. The green compacts were sintered at 1600°C for 2 hours. The fired specimens were machined with #600 resin-bonded diamond wheel at cutting depth of 1μm/pass. Equal stock were removed from both the test surface, tension (support) and compression (loaded). The final dimensions of the disc specimens were about 43mm in diameter and 2.2mm thick. Forty disc specimens were manufactured from the same powder lot and were prepared at the same condition.

4.3.4 Testing jig designs

The test jig used in the work is shown in Fig. 4.6. The jig was designed to have a 30mm supported ring diameter. The ring was made from die steel (hardened and tempered) and top-surface of the ring is radiused to 5mm. The ball used to apply the load was made from a high carbon chrome alloy steel (AISI 52100) and had a diameter of 5mm and 10mm, respectively. The most important consideration in the design was that there should be no eccentricity of loading and the self-aligning of the planes must be achieved.

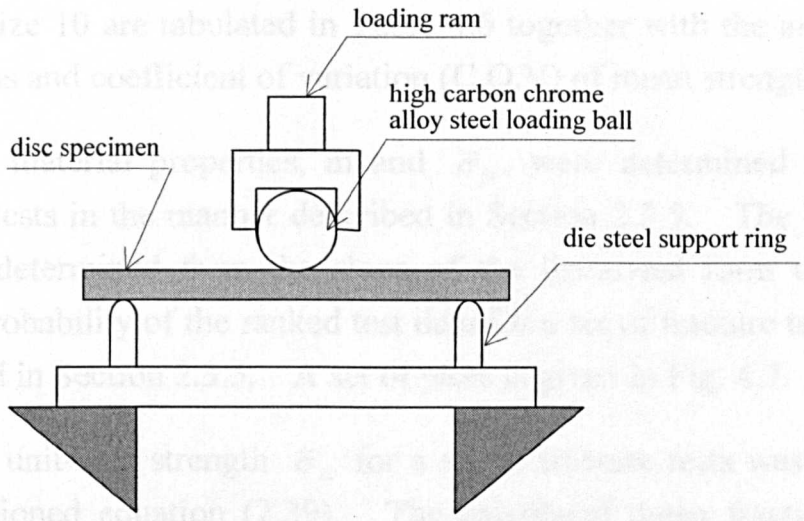


Fig. 4.6 Schematic of the ball-on-ring test jig

4.3.5 Determination of material properties

The strength of the disc specimens was determined by ball-on-ring testing at ambient room-temperature conditions with the same desktop testing machine (CK10) described in Section 4.2.5.

For cost reasons, a set of only ten pieces of disc specimens was tested. Each disc specimen was centralized in the jig relative to the support. All specimens were loaded to fracture on the same CK 10 testing machine with 30mm support ring diameter at a loading rate of 0.5 mm/min. The load ball size used in this ball-on-ring test was 10mm in diameter.

All stresses were computed from the mentioned equations (4.2) and (4.3). No data were to be discarded. The values of Poisson's ratio assumed in equations (4.2) and (4.3) were 0.26 and 0.3 for the disc plate and steel ball, respectively; while Young's moduli for disc specimen and ball were assumed to be 380 GPa and 205 Gpa, respectively.

The individual fracture loads and fracture strength, σ_f, for discs in ball-on-ring testing are given in Table 4.5. The mean strength, $\bar{\sigma}_f$, for a set of tests of sample size 10 are tabulated in Table 4.6 together with the associated standard deviations and coefficient of variation (C.O.V) of mean strength.

The material properties, m and $\bar{\sigma}_{fa}$, were determined from each set of fracture tests in the manner described in Section 2.3.5. The Weibull modulus, m, was determined from the slope of the linearised form of the cumulative failure probability of the ranked test data for a set of fracture tests in the manner described in Section 2.3.5. A set of plots is given in Fig. 4.7.

The unit-area strength $\bar{\sigma}_{fa}$ for a set of fracture tests was determined from the mentioned equation (2.29). The calculated mean fracture load, Weibull modulus and unit-area strength for a set of fracture tests is given in Table 4.7.

SpecimenNo.	Specimen dia. (mm)	Specimen thickness (mm)	Fracture load (N)	Fracture strength (MPa)
K1	43.61	2.21	1051	588
K2	43.56	2.16	974	575
K3	43.68	2.18	984	569
K4	43.37	2.13	974	591
K5	43.49	2.23	1024	565
K6	43.52	2.27	1002	534
K7	42.52	2.18	825	484
K8	42.80	2.15	882	530
K9	43.77	2.22	1070	593
K10	42.31	2.25	798	441

Table 4.5 The fracture loads and fracture strength for discs in ball-on-ring testing.

Test model	Mean strength (MPa)	Standard deviation (MPa)	Coefficient of variation (%)
ball-on-ring test support ring dia.=30mm load ball dia.=10mm loading rate = 0.5mm/min	547	51	9.3

Table 4.6 The mean strength for a set of ball-on-ring tests

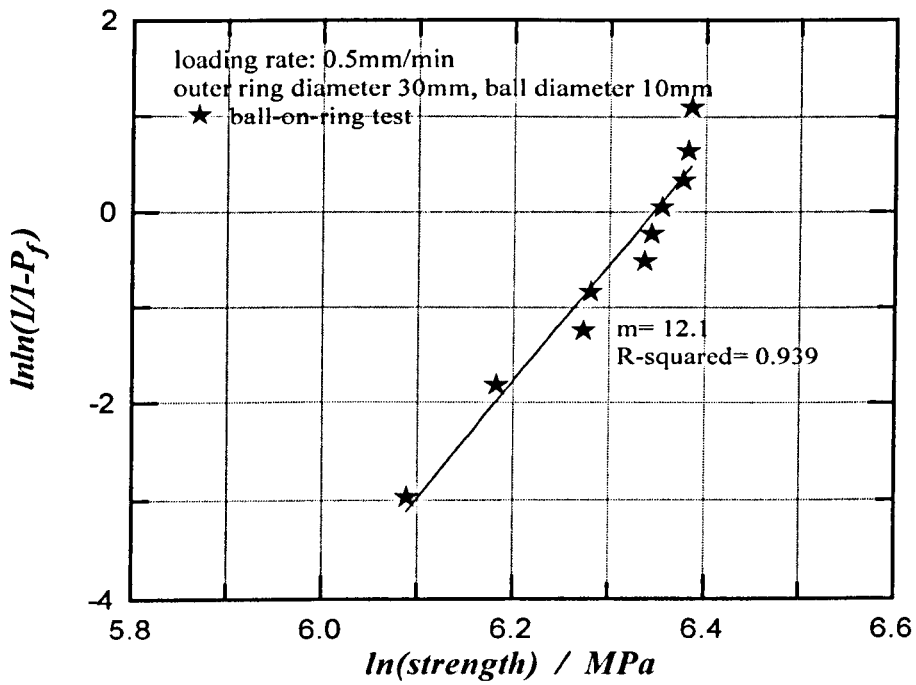


Fig.4.7 The Weibull curve for the ball-on-ring test

Test model	Mean fracture load (N)	Weibull modulus	Unit-area strength (MPa)
ball-on-ring test support ring dia.=30 mm load ball dia.=10 mm loading rate = 0.5mm/min	958	12.1	361

Table 4.7 The unit-area strength for a set of ball-on-ring tests

4.3.6 The effects of load ball diameter and the nature of the support and load on fracture strength

In order to investigate the effect of testing parameters of ball-on-ring test on the determination of the fracture strength, a series of tests were carried out. A ball-on-ring test jig with supported ring of 30mm diameter was set up on a CK 10 testing machine.

Twenty disc specimens were tested to evaluate the influence of load ball diameter. The ball-on-ring tests were performed using a CK 10 test machine on these disc specimens at a loading rate of 0.5 mm/min with various loading ball diameter from 10mm to 5mm. Ten pieces of specimen were tested for each loading ball diameter.

Another ten discs were tested by ring-on-ring testing to evaluate the effect of varying the nature of the load for the two test methods (ring-on-ring and ball-on-ring) for the same support ring diameter.

The other ten discs were tested by 4-Ball testing (See Section 4.4) with same loading ball diameter and support diameter as that used in ball-on-ring testing for the evaluation of the dependence of the nature of support.

The fracture results were analysed as before (Section 4.3.5). The individual fracture results are given in Appendix 6. The estimated parameters

of this investigation are given in Table 4.8 and shown in Fig. 4.8, 4.9 and 4.10.

The following is a summary of the main points obtained from the preceding experiments:

(1) The mean fracture strength obtained from ball-on-ring test using a 5mm load ball diameter was slightly greater than that obtained using a 10mm ball diameter. The coefficient of variation of testing results obtained from a 5mm ball diameter was smaller than that obtained from a 10mm ball diameter.

(2) For the same support ring diameter and same loading rate the mean fracture strength obtained from the two test methods (ring-on-ring and ball-on-ring) is dependent of the nature of the load. The disc specimens loaded by a ball were found to have larger fracture strength and a smaller coefficient of variation than that loaded by a ring.

(3) For the same support diameter and same load ball diameter, the mean fracture strength obtained from the two test methods (ball-on-ring and 4-Ball) is independent of the nature of the support.

The experimental results have shown that the load ball diameter did slightly influence the fracture strength in the ball-on-ring test and that the mean fracture strength is dependent of the nature of the load but independent of the nature of the support. It is known that the observed strength value is dependent on the flaw size distribution of the material and the stress distribution in the test specimen. The greater mean fracture strength as mentioned in the preceding conclusions may be ascribed to the smaller area and volume of material under peak tensile stress. As for the comparison of the coefficient of variation, the narrower distribution of flaw size in the specimen volume is the main reason to cause its smaller value of the coefficient of variation.

Model No.	Mean strength (MPa)	Standard deviation (MPa)	Coefficient of variation (%)	Weibull modulus
1 ball-on-ring test support ring dia.=30mm load ball dia.=10mm loading rate=0.5 mm/min	547	51	9.3	12.1
2 ball-on-ring test support ring dia.=30mm load ball dia.=5mm loading rate=0.5 mm/min	557	25	4.5	26.0
3 ring-on-ring test support ring dia.=30mm load ring dia.=10mm loading rate=0.5 mm/min	296	47	15.9	7.4
4 4-Ball test pitch circle dia.=30mm load ball dia.=5mm loading rate=0.5 mm/min	555	43	7.7	14.6

Table 4.8 The effect of testing parameter in a ball-on-ring test

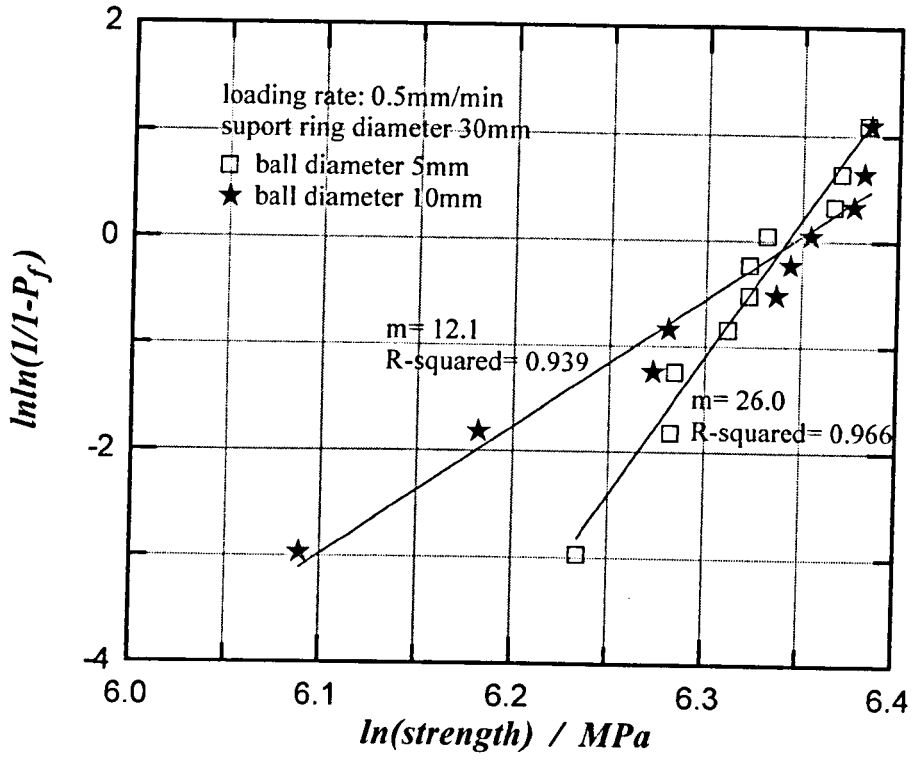


Fig. 4.8 The Weibull curves for the ball-on-ring test using various ball diameters

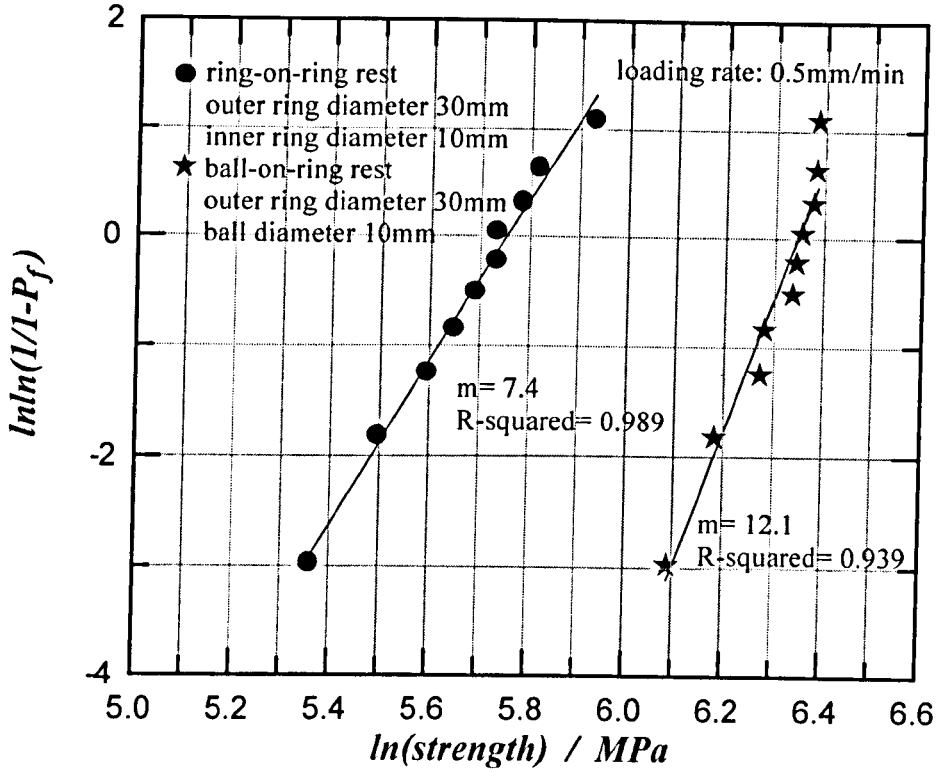


Fig. 4.9 The Weibull curves for the ball-on-ring test and the ring-on-ring test

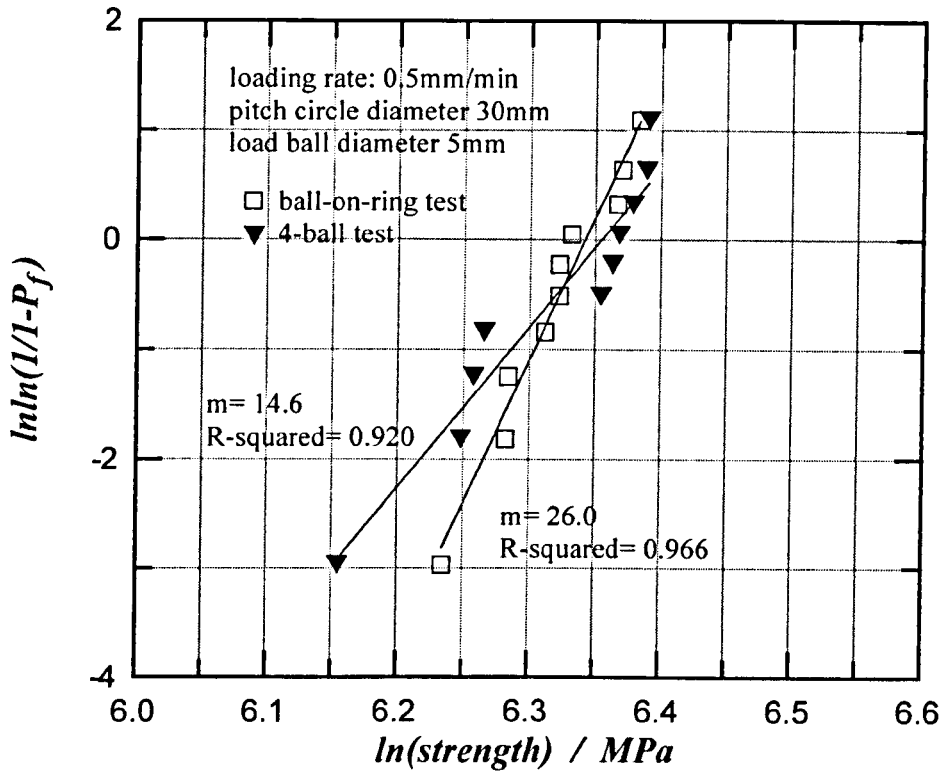


Fig. 4.10 The Weibull curves for the ball-on-ring test and 4-Ball test

4.4 The 4-Ball test

4.4.1 Introduction

The 4-Ball test involves a ball-loaded disc supported by three equi-spaced balls. It possesses attractive features which include the following [13]:

1. The disc specimens are easy to produce.
2. The test is not necessarily critically affected by poor specimen tolerance.
3. The test is simple to perform.
4. Specimen failure is independent of edge conditions (in contrast to beam specimens).

In addition to the above, the 4-Ball test is more attractive than the ring-on-ring test in cases where the disc specimen surfaces are warped or slightly

irregular. The 4-Ball test has much in its favour since contact with all four balls is assured.

D. J. Godfrey [86] used this test at the Admiralty Research Establishment, UK, to examine Si_3N_4 ceramic formulations for demanding applications, and to characterize properties and environmental effects such as oxidation, corrosion and chemical attach and contamination.

In the following, the stress analysis of the 4-Ball test is demonstrated. The test specimen and testing jig designs are described. The fracture strength and Weibull modulus of alumina ceramics subjected to a series of 4-Ball test are presented. The effect of the loading rate, ball diameter and pitch circle diameter on fracture strength are also evaluated.

4.4.2 Stress analysis

The 4-Ball test has no known exact stress solution. As a first attempt at stress analysis, Sivill [12] developed Timoshenko's [81] circular plate equations for a ring-supported plate loaded with uniform pressure acting over a small region near the centre. The derivation of the analytical solution for the deflection, bending moment and stresses for this plate subjected a uniformly distributed pressure load are given in Appendix 7 [13].

Consider a thin circular disc, simply supported and loaded as shown in Fig. 4.11:

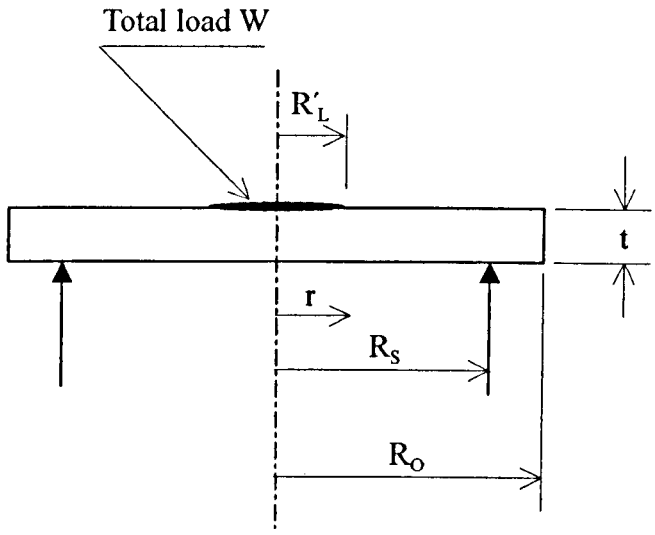


Fig. 4.11 The ball-loaded ball-supported disc in bending

the maximum bending stress obtained occurs at the centre and is shown as

$$\sigma_{max} = (3W/2\pi t^2)\,[(1+\nu)\ln(R_S/R'_L) + (1+\nu)/2 + (1-\nu)\,(2R_S^2 - R'_L)/4R_O^2] \quad (4.4)$$

where R'_L is the "contact radius" of the loading ball given by Roark [85] as

$$R'_L = 0.721[Wd((1-\nu_b^2)/E_b + (1-\nu^2)/E)]^{1/3} \quad (4.5)$$

where d : ball diameter

E_b: Young's moduli of the ball

E : Young's moduli of the disc plate

ν_b: Poisson's ratio of the ball

ν : Poisson's ratio of the disc plate

W: applied load

t : thickness of the disc plate

R_S: radius of pitch circle diameter

R_O: radius of the disc plate

Hence the tensile fracture strength, σ_f (i.e. the maximum stress on the lower surface of the disc at fracture), can be estimated from equations (4.4) and (4.5) if the fracture load W_f, the Young's moduli and Poisson's ratio of the ball and disc materials are known or values assumed for them.

However, the stress-volume integrals derived from these analytical stresses will be in error because [12]:

1. Thin plate equations have been used and this specimen geometry would clearly require a thick plate treatment.
2. The solution will be imperfect under the load at which point the stress is greatest.
3. Uniformly distributed loading is not an accurate representation of ball loading.
4. Friction at the contacts is neglected.

Some of these points could almost certainly be overcome by developing a more exact analytical solution. First, the finite element method was examined for its suitability for stress determination in this case. For the 4-Ball test either pure bending of every section can be assumed or three dimensional elements must be used. Pure bending elements prove unsatisfactory for such a thick section; three dimensional elements were not used because of the computer limitation on a number of elements. Therefore, the finite element method was not adopted for the stress analysis of the 4-Ball test. So an experimental approach was adopted for the more accurate stress analysis.

Karroum [13] carried out a complete photoelastic stress analysis for the stress determination of the 4-Ball and ball-on-ring test. Analysis of the results confirmed the close similarity between the high stresses for the two loading tests for the same support diameter. A measurement of the central deflection relative to the support also showed that the deflections for the two tests, for the same support diameter, agree to within 3%. An examination of the isochromatic fringe patterns through a polariscope, further confirmed the close similarity of the high stresses near the centres of the discs. Since the stress integrals are strongly dependent on these high stresses it is assumed that the same stress-volume and stress-area integrals apply for the two tests.

4.4.3 The test specimen

The specimens were manufactured by die-pressing an alumina powder (99.5% Al_2O_3, AES-11, Sumitomo Chemical Co., Ltd., Japan) onto a disc plate. The preparation and surface finish of disc specimens were the same as in the ring-on-ring testing. The pressing pressure employed was 196 MPa. The green compacts were sintered at 1600°C for 2 hours. The fired specimens were machined with #600 resin-bonded diamond wheel at a cutting depth of 1 μm/pass. Equal stock were removed from both the test surface, tension (support) and compression (loaded). The final dimensions of the disc specimens were about 43mm in diameter and 2.2mm thick. Fifty disc specimens were manufactured from the same powder lot and were prepared at the same conditions.

4.4.4 Testing jig designs

The testing jig used in this study of the 4-Ball test is shown in Fig. 4.12. The jig was designed to have a 30 mm and a 40 mm pitch circle diameter of equi-spaced supporting balls. The basic plate design for the 4-Ball test was shown in Fig. 4.13. The four balls (the loading ball and three supporting balls) in any one 4-Ball test were identical. The balls used in this work were made from high carbon chrome alloy steel (AISI 52100) and had a diameter of 5mm and 10mm respectively. The most important consideration in the design was that there should be no eccentricity of loading.

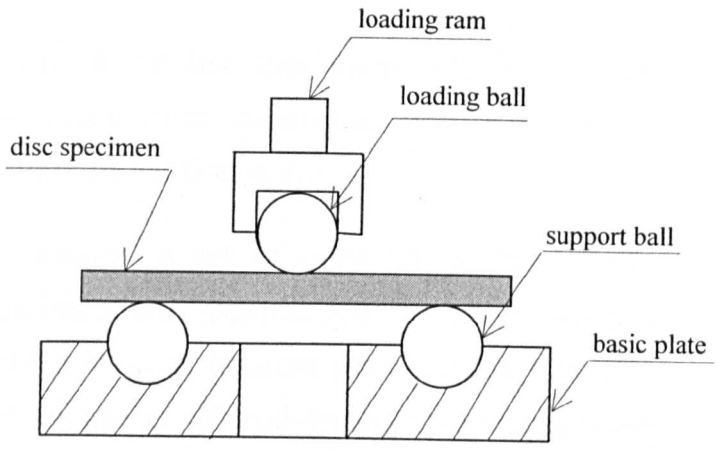


Fig. 4.12 Schematic of the 4-Ball test jig

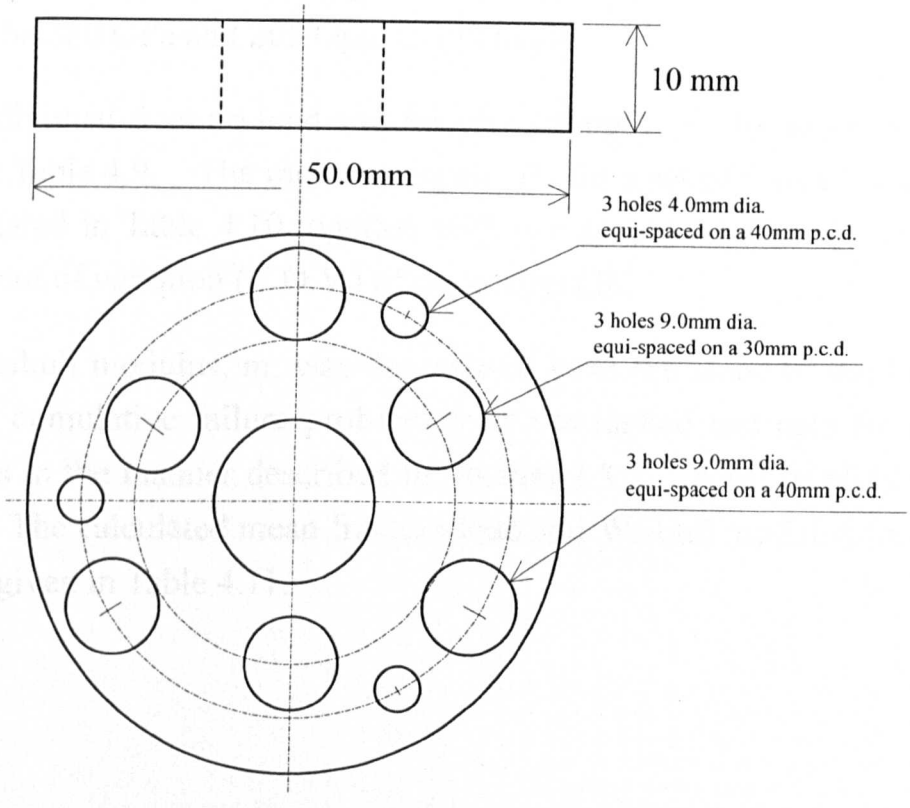


Fig. 4.13 The basic plate design for 4-Ball test

4.4.5 Determination of material properties

The strength of the disc specimens was determined by the 4-Ball testing at ambient room-temperature conditions with the same desktop testing machine (CK 10) described in Section 4.2.5.

For cost reasons, a set of only ten pieces of disc specimen was tested. Each disc specimen was centralized in the jig relative to the support. All specimens were loaded to fracture on the same CK 10 testing machine with 30 mm pitch circle diameter of equi-spaced supporting balls at a loading rate of 0.5 mm/min. The ball size used in this 4-Ball test was 10mm in diameter.

All stresses were computed from the mentioned equations (4.4) and (4.5). No data were to be discarded. The values of Poisson's ratio assumed in equations (4.4) and (4.5) were 0.26 and 0.3 for the disc plate and steel ball bearing, respectively; while Young's moduli for disc specimen and ball were assumed to be 380 GPa and 205 Gpa, respectively.

The individual fracture load and fracture strength σ_f, for discs in bending are given in Table 4.9. The mean strengths $\overline{\sigma}_f$ for a set of tests of sample size 10 are tabulated in Table 4.10 together with the associated standard deviations and coefficient of variation (C.O.V.) of mean strength.

The Weibull modulus, m, was determined from the slope of the linearised form of the cumulative failure probability of the ranked test data for a set of fracture tests in the manner described in Section 2.3.5. A set of plots is given in Fig. 4.14. The calculated mean fracture load and Weibull modulus for a set of fracture are given in Table 4.11.

Specimen No.	Specimen dia. (mm)	Specimen thickness (mm)	Fracture load (N)	Fracture strength (MPa)
A1	43.24	2.16	971	573
A2	43.24	2.18	935	543
A3	43.24	2.16	904	536
A4	43.24	2.18	930	541
A5	43.16	2.15	981	584
A6	43.32	2.16	935	553
A7	43.20	2.16	969	572
A8	43.22	2.20	1041	589
A9	43.24	2.20	1003	569
A10	43.24	2.19	976	560

Table 4.9. The Fracture loads and fracture strength for disc in 4-Ball testing

Test model	Mean strength (Mpa)	Standard deviation (Mpa)	Coefficient of variation (%)
4-Ball test pitch circle dia.= 30 mm ball dia.= 10 mm loading rate = 0.5mm/min	562	18	3.2

Table 4.10 The mean strength for a set of 4-Ball test

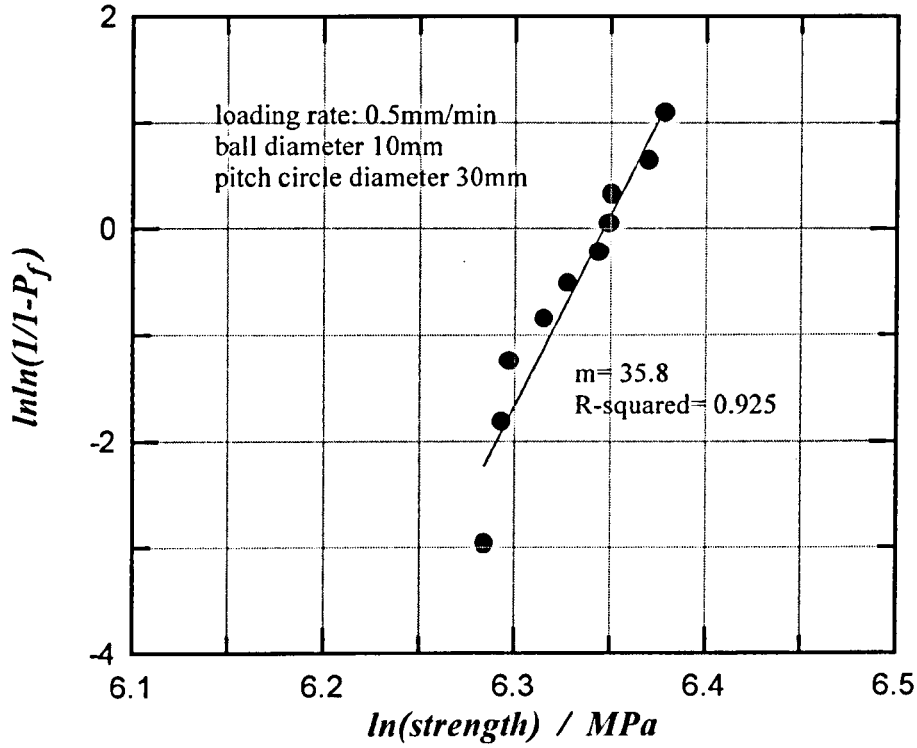


Fig 4.14 The Weibull curve for the 4-Ball test

Test model	Mean fracture load (N)	Weibull modulus
4-Ball test pitch circle dia.=30mm ball dia.=10mm loading rate=0.5 mm/min	965	35.8

Table 4.11 The Weibull modulus for a set of 4-Ball test

4.4.6 The effects of the loading rate, ball diameter and pitch circle diameter on fracture strength

In order to investigate the effect of testing parameters on the determination of the fracture strength, a series of the 4-Ball test were carried out. A 4-Ball test jig with a 40mm and a 30mm pitch circle diameter was set up on a CK 10 testing machine. A set of fifty pieces of disc specimen was prepared.

Thirty discs were tested to evaluate the influence of loading rate. The 4-Ball test with a 30mm pitch circle diameter and a 10mm ball diameter were performed using a CK10 test machine on these disc specimens with various loading rates of 0.5, 0.05, and 2 mm/min, respectively. Ten specimens were tested for each loading rate.

Another ten discs were tested to evaluate the pitch circle distance effect by varying the pitch circle diameter from 30mm to 40mm.

The other ten discs were tested for the evaluation of ball diameter dependence of fracture strength by varying the ball diameter from 10mm to 5mm.

The fracture results were analysed as before (See Section 4.3.5). The individual fracture results are given in Appendix 8. The estimated parameters of this investigation are given in Table 4.12 and shown in Fig. 4.15, 4.16 and 4.17.

The following is a summary of the main points obtained from the preceding experiments:

(1) The loading rate did strongly influence the fracture strength. For the same pitch circle diameter and same ball diameter, the slower the speed of loading, the smaller the mean fracture strength obtained. However, the loading rate of 0.5 mm/min was found to have the smallest value of coefficient of variation.

(2) The mean fracture strength obtained from the 4-Ball test using a 30mm pitch circle diameter did not differ significantly from that obtained using a 40mm pitch circle diameter. However, the coefficient of variation of

testing results obtained from a 30mm pitch circle diameter was smaller than that obtained from a 40mm pitch circle diameter.

(3) The mean fracture strength obtained using 5mm ball diameter was slightly greater than that obtained using a 10mm ball diameter. The mean fracture strength values from the two sets of test differed by only 2.7%. The coefficient of variation of testing results obtained from a 5mm ball diameter was smaller than that obtained from a 10mm ball diameter.

The rate of loading can have an effect on fracture strength as the result of stress corrosion mechanism, particularly at low strain rates. In general, the slower the rate of loading, the greater the opportunity for stress corrosion phenomena to weaken the specimen.

The experimental results have shown that the pitch circle diameter of the test jig did not strongly influence the fracture strength. With the same load diameter and same loading rate, the area and volume of material under peak tensile stress was similar for the 4-Ball test using 30mm pitch circle diameter and 40mm pitch circle diameter, and thus the measured strength did not differ significantly.

The surface area and volume of material under 5mm load ball is smaller than that under 10mm ball, therefore, there are smaller area and volume of material under peak tensile stress in the 4-Ball test using a 5mm ball. This leads to the greater fracture strength obtained from a 5mm ball.

As for the comparison of the coefficient of variation mentioned in conclusions (2) and (3), the narrower distribution of flaw size in the specimen volume under 30mm pitch circle diameter or 5mm ball is the main reason to cause its smaller value of the coefficient of variation.

Model No.	Mean strength (MPa)	Standard deviation (MPa)	Coefficient of variation (%)	Weibull modulus
1 pitch circle dia.=30mm ball dia.=10mm loading rate=0.5 mm/min	562	18	3.2	35.8
2 pitch circle dia.=30mm ball dia.=10 mm loading rate=0.05mm/min	510	23	4.5	25.2
3 pitch circle dia.=30mm ball dia.=10mm loading rate=2mm/min	612	23	3.8	30.3
4 pitch circle dia.=40 mm ball dia.=10 mm loading rate=0.5 mm/min	555	29	5.2	22.2
5 pitch circle dia.=40 mm ball dia.=5 mm loading rate=0.5 mm/min	570	23	4.0	28.8

Table 4.12 The effect of testing parameters in 4-Ball test

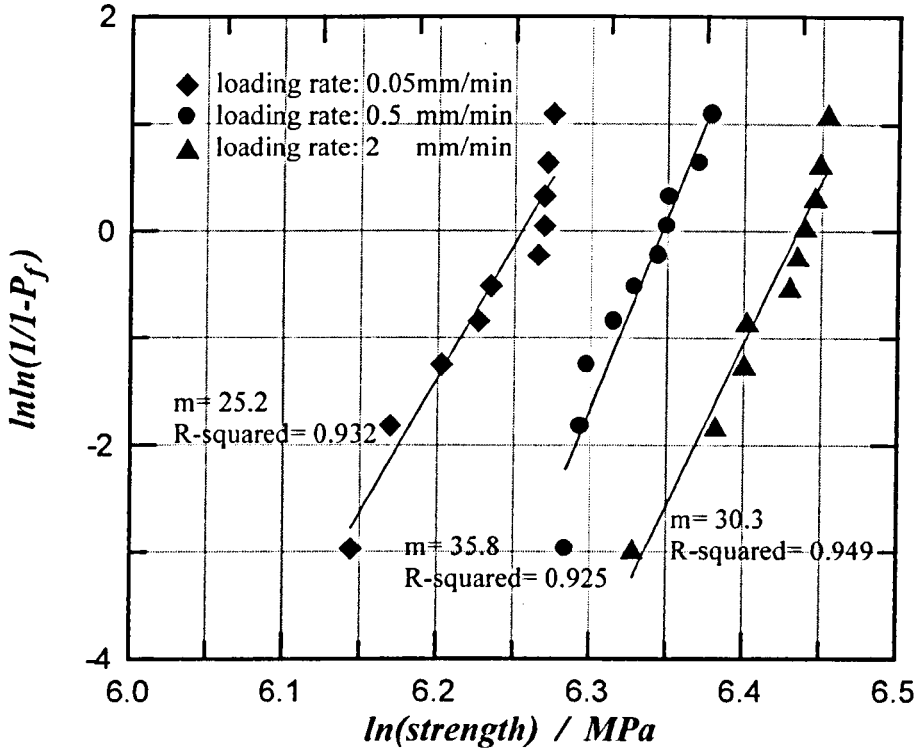


Fig. 4.15 The Weibull curves for the 4-Ball test using various loading rates.

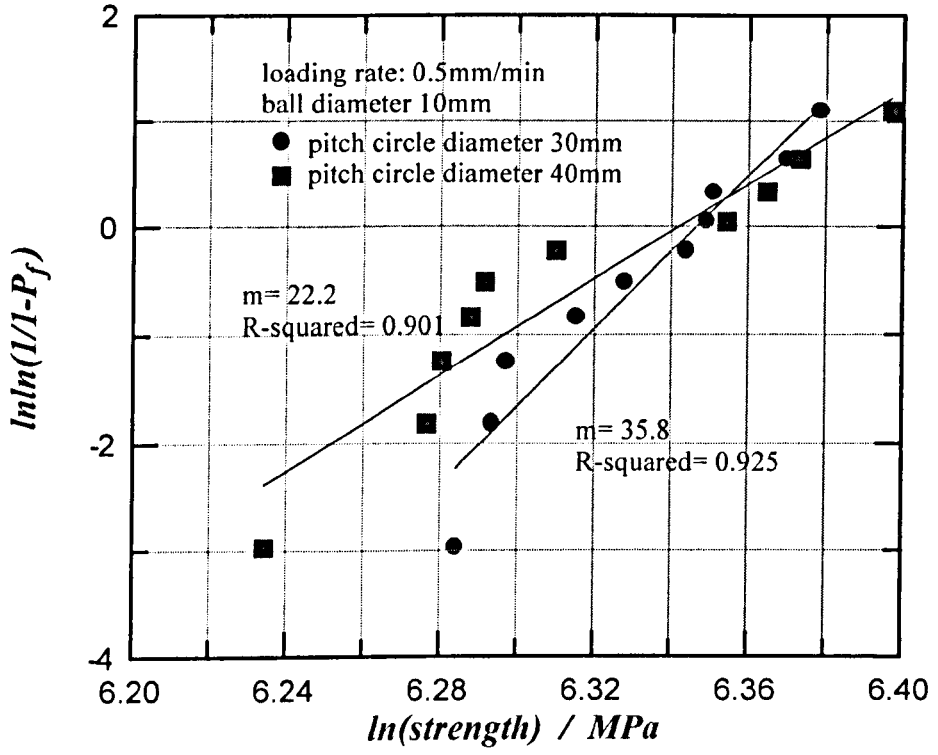


Fig. 4.16 The Weibull curves for the 4-Ball test using various pitch circular diameters.

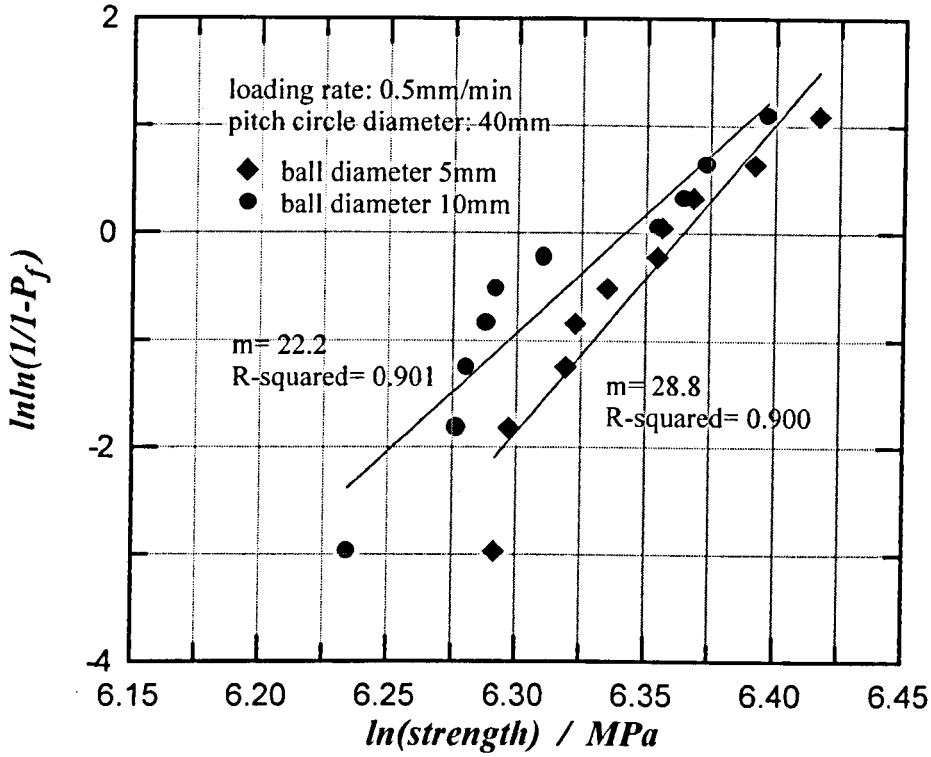


Fig. 4.17 The Weibull curves for the 4-Ball test using various ball diameters.

4.5 Summary

Measuring the strength of engineering ceramics by the biaxial flexure tests possesses attractive features which include the following:

(1) The conventional uniaxial beam bending test is often of limited value for the design engineer since the loading under service conditions is seldom of pure bending, compression or torsion, respectively. Therefore, the biaxial flexure test on discs with biaxial stresses is advisable.

(2) The disc specimens for the tests are easy to produce.

(3) The tests are simple to perform.

(4) The tests are not critically affected by poor specimen tolerance.

(5) It is difficult to separate edge and surface effects on the measured strength in the uniaxial flexure tests. Nevertheless, specimen failure in the biaxial

flexure test would not be dependent upon edge conditions.

There are many possible ways for the biaxial flexure tests. Six such test methods are the ring-loaded ring-supported (ring-on-ring test), the ball-loaded ring-supported (ball-on-ring test), the ball-loaded disc supported by three equispaced balls (4-Ball test), the piston-loaded ring-supported (piston-on-ring test), the piston-loaded disc supported by three equispaced balls (piston-on-3-ball test) and hydraulic pressure loading of discs (hydraulic pressure test).

The theoretical analysis and experimental investigation of three major techniques of the biaxial flexure tests for ceramics, i.e. ring-on-ring, ball-on-ring and 4-Ball tests were described. The effects of varying the test parameters were also discussed.

The ring-on-ring test involves supporting a circular plate on a ring and loading with a small concentric ring. The ring-on-ring loading fixture was constructed to provide a biaxial-tension-strength test in which the effective stressed area or volume of the specimen is comparable to the conventional uniaxial-flexure tests, such as four-point beam bend tests. The test is gaining considerable popularity, as an exact analytical stress solution is available for it. In the experimental investigation of the effects of testing parameters on the strengths, it was found that the fixture size of the test jig did strongly influence the fracture strength. For the same loading rate, the larger the outer ring diameter to inner ring diameter ratio, the greater the mean fracture strength obtained. The test with an outer ring diameter of 40 mm and an inner ring diameter of 10 mm showed the smallest value of coefficient of variation.

The ball-on-ring test involves supporting a disc plate on a ring and centrally loading with a ball. Its advantages are precise knowledge of the stresses produced in the specimen, simple test fixtures and specimen geometry, and minimum requirements for alignment. The following is a summary of the main points obtained from the experimental investigation of the effect of testing parameters on the determination of the fracture strength:

(1) The load ball diameter did slightly influence the fracture strength. The mean fracture strength obtained from ball-on-ring test using a 5 mm load ball diameter was slightly greater than that obtained using a 10mm ball

diameter. However, the coefficient of variation of testing results obtained from a 5mm ball diameter was smaller than that obtained from a 10mm ball diameter.

(2) For the same support ring diameter and same loading rate, the mean fracture strength obtained from the two test methods (ring-on-ring and ball-on-ring) is dependent of the nature of the load. The disc specimens loaded by a ball was found to have larger fracture strength and smaller coefficient of variation than that loaded by a ring.

(3) For the same support diameter and same load ball diameter, the mean fracture strength obtained from the two test methods (ball-on-ring and 4-Ball) is independent of the nature of the support.

The 4-Ball test involves a ball-loaded disc supported by three equi-spaced balls. The three-ball support is advantageous because it provides kinematic mounting for a flat disc specimen. Kinematic mounting cannot easily be achieved when more than three balls are used, which could lead to spurious results. The 4-Ball test is more attractive than the ring-on-ring test, in cases where the disc specimen surfaces are warped or slightly irregular. In the experimental investigation of the effect of testing parameters on the fracture strength, the main points obtained can be summarized as:

(1) The loading rate did strongly influence the fracture strength. For the same pitch circle diameter and same ball diameter, the slower the speed of loading, the smaller the mean fracture strength obtained. However, the loading rate of 0.5mm/min was found to have the smallest value of coefficient of variation.

(2) The pitch circle diameter of test jig did not strongly influence the fracture strength. The mean fracture strength obtained from the 4-Ball test using a 30mm pitch circle diameter did not differ significantly from that obtained using a 40mm pitch circle diameter. However, the coefficient of variation of testing results obtained from a 30mm pitch circle diameter was smaller than that obtained from a 40mm pitch circle diameter.

(3) The ball diameter did slightly influence the fracture strength. The mean fracture strength obtained using a 5mm ball diameter was slightly greater

than that obtained using a 10mm ball diameter. The mean fracture strength values from the two sets of tests differed by only 2.7%. The coefficient of variation of testing results obtained from a 5mm ball diameter was smaller than that obtained from a 10mm ball diameter.

The observed strength value is dependent on the type of test conducted. More specifically, it is dependent on the flaw size distribution of the material and the stress distribution in the test specimen. As the uniformity of the flaws within a material increases, the strength values measured approach each other.

CHAPTER 5
Development of Testing Method Standards

5.1 Introduction

As mentioned earlier, there are many reasons to establish standards for engineering ceramics. One is the creation of a common language. Standards are the international language of science and engineering. On the basis of standards, industrial suppliers and their customers around the world can reach assured understanding about products and their performance, another need is to address concerns of public health and safety, where appropriate, this includes the impact on the environment. The primary need may lie in the assurance of quality and performance that is provided by standards.

For the future outlook of the engineering ceramics industry, the need for standards will increase substantially. Firstly, the ever wider use of ceramic materials and the development of hybrid forms confront users with increasingly complex problems in their choice of material. It is only by establishing suitable standards, and in particular by defining at least rudimentary classifications, users can be helped to select materials. Secondly, the increased stringency of quality requirement of products calls for closer control of production processes. Standard test methods can be used for monitoring the quality of products during the intermediate manufacturing phase. Finally, the broadening of the industrial base and progress in bio-medical research will continue to generate increased legislation on health safeguards and pollution control. If there are no economic incentives to use clean processes, it is expected to have recourse to standardisation.

Standardisation activities in the United Kingdom are directed along three main lines: the development of standard test methods, the production of standard specifications, and the development of regulatory codes. The Collyear Report in the United Kingdom stressed, some years ago, the importance of test methods standardisation. The development of testing method standards has been the focus of much attention recently.

Measurement of flexural strength must be accurate if they are to be really useful and reliable. In order to obtain more consistent and accurate test results, the strandardisation of flexure testing of engineering ceramics must be carried out. Since the uniaxial flexure tests have been standardised by the JIS, ASTM, DIN, ANFOR, BSI, and EN etc., the development of standards for biaxial flexure test method which can be easily performed and possess the accurate and consistent flexure testing results becomes the ultimate objective of the work.

In this chapter, the methodology for the formulation of the document standard is developed. The drafts of standards for three major techniques of the biaxial flexure tests for ceramics, i.e. ring-on-ring, ball-on-ring, and 4-Ball tests are presented. The main points considered during the preparation of these standards are also discussed.

5.2 Methodology

In respect of the procedures and practices adopted for the formulation of standards, there exists some degree of diversity, but a much greater degree of uniformity is quite evident. The typical procedures for developing a document standard may be grouped into the following four stages: justification of proposals, preparation of drafts, modification of drafts, and approval and publication of standards. Generally speaking, the minimum normal time for the creation of a standard is one and a half years, corresponding to the adaptation of a preexisting standard. Standards that are wholly new or are major revisions have required two to four years to adopt.

5.2.1 Principles for formulation of standards

Standard should be wanted, used, and planned as mentioned earlier. The basic principles for the formulation of standards can be summarized as:

1. Standards shall fulfil a generally recognized need of industry, trade, technology and other sectors of human life.
2. Standards shall represent the largest possible consensus of opinion between all the interests concerned.

3. Standards shall keep in view the latest scientific advancements, but remain technologically and economically practical for application to various sectors of activity to which they relate.
4. Standards shall be so designed as to encourage the development of more efficient economical practices, and leave the way open for devising new ways and means for carrying out operations more efficiently and effectively.
5. Standards shall be subject to periodic revision and amendment and be kept up-to-date in respect of the latest advancements of technology and the changing circumstances.
6. Standards shall safeguard the interests of both the producer and the consumer.
7. Standards shall be in accordance with the current and immediate future needs of the economy of the country.
8. Standards shall be prepared at the broadest level consistent with meeting the needs of interested parties within an acceptable time-scale.
9. Standards shall be written in a simple and clear way.

5.2.2 Procedures of preparation of standards

The principles enunciated in the foregoing section would provide the guidelines for laying down the procedures for the preparation of standards. Generally speaking, the procedures would include the following stages. The flow chart of the procedures is outlined in Fig.5.1.

1. Justification of proposals

This stage involves the emergence and receipt of proposals, preliminary scrutiny of proposals, and approval of the projects. Proposals for preparing new standards or revising or amending existing standards may arise from the needs of any sector of economy. The major authentic sources are:

(1) Organizations of industry, trade, technologists, consumers and users.

(2) Individual industrial units, commercial houses, technologists, professional engineers, industrial and other users and consumers.

(3) Government departments and agencies.

(4) Councils, committees, sub-committees and panels of standards body or their constituent members.

(5) Any other organized body having an interests in standardisation in the proposed field.

Each one of the proposals for new work received in this manner is first of all examined by the secretariat of standards body to determine:

(1) Whether the proposal is such as may be considered consistent with the principles enunciated above.

(2) Whether related standards in the field already exist.

(3) Whether entirely original standard would have to be prepared.

(4) Whether any survey, investigation or research work would have to be undertaken.

(5) Whether in making a decision for approval of the project it would be necessary to call a conference of the interests concerned, or to make a postal enquiry among such interests, or whether the relevant division council or industry committee could perhaps act directly.

(6) Whether a competent technical or sectional committee exists which could be allotted the work, if it were approved, or whether a new committee would have to be created.

The result of the preliminary scrutiny carried out by the secretariat of standards body, together with its recommendation for further action, is placed before the divisional council or the industry committee concerned for deciding whether the proposal be approved and a project for new work set-up, or rejected or its consideration postponed.

2. Preparation of drafts

In case of proposal is decided to be approved, the work is allotted to an existing technical committee or sectional committee. On due allotment of the subject, the technical committee proceeds with the preparation of draft standards. This stage is by far the most important and most time-consuming element of the

whole procedure. The success or failure of any standardisation project would depend on how well this part of the work is organized and carried out. When component personnel is available among committee members to undertake the task of drafting, it is often convenient for the technical committee to put them together in a subcommittee or a panel so that they could divide the work among themselves according to their own convenience.

When the data available in standards from outside sources are inadequate or unsatisfactory for the purpose of drafting standards, it becomes necessary to gather original data. In such a case the committee or subcommittee responsible may initiate action along any one or more of the following lines of approach whichever is appropriate:

(1) Issue a questionnaire to all the known interested parties to collect the necessary information.

(2) Initiate surveys, investigations and research projects in cooperation with institutions are competent and adequately equipped and manned for the purpose.

3. modification of drafts

This stage includes the wide circulation of drafts, compilation of comments, and finalization of drafts. Upon completion of the draft standard, it should be publicly circulated for several months to give knowledgeable persons and bodies a chance to prepare a submission of their views about the draft. The object of wide circulation is to inform every interest that may be affected about the contents of the draft and invite their critical review and comments, with a view to modifying it suitably in such a way as to make it more generally acceptable. Normally, the secretariat of standards body arranges for copies of the draft to be sent to as many concerned parties and invites their close study and suggestions.

The comments received as a result of wide circulation should be so collected and collated that on presentation to the technical committee could be systematically examined and corresponding decisions conveniently recorded. In taking decisions on the various comments, the technical committee will again have to rely on the basic principles enunciated above but it must always keep in

view the desirability of adopting such compromises as would enable maximum possible implementation of the standard to be secured on its publication. In the light of technical committee decisions, the final version of the draft is compiled by the secretariat for the next stages of processing, which are largely formal.

4. Approval and publication of standards

The final version of the draft is now ready for being accepted as the formal standard. The procedures for this acceptance vary in different standards body. But wherever there exist division councils or industries committee, the draft is first presented to the relevant body for acceptance or, on behalf of the body concerned, to its chairman. Then it goes to the supreme body, or its chairman on its behalf, for final approval as a formal standard, and then it is published.

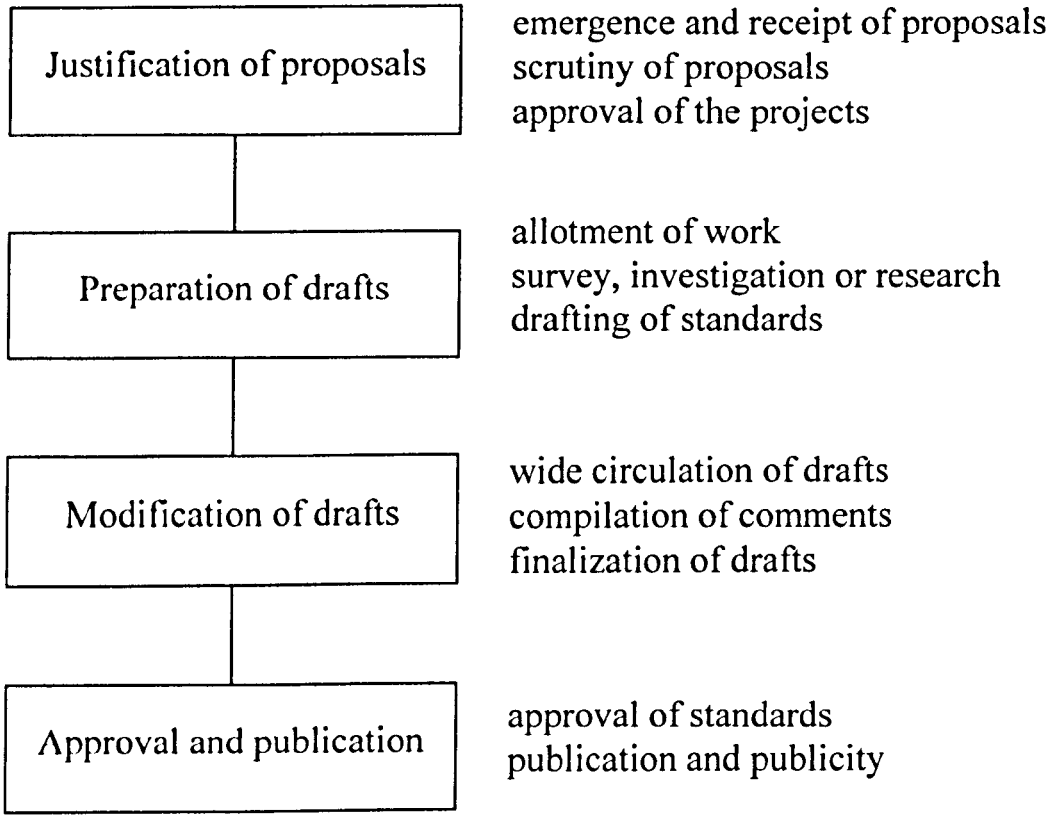


Fig. 5.1 Flow chart of the procedures for the preparation of standards

5.2.3 Strategy for formulation of standards

With the increase in usage of advanced materials and products, a new industrial standard is required in order to evaluate properties of them and to perform fair and just trade. Because of this essential function, the enactment of industrial standardisation has to follow the practical usage of advanced materials. In other words, the important role of standardisation for the advanced materials is known after the practical and common usage of them. This conventional way of determination for standards sometimes does not work well on recent advanced materials like engineering ceramics.

The practical usage of advanced materials is established over a long period after many physical and chemical tests, this pertains especially for structural applications. This is because structural applications require not only the satisfaction of needed physical and chemical properties but also the long-term reliability of the materials. This delay of practical usage will obstruct opportune enactment of industrial standards for new materials if the conventional way of determination for standards as described above obeyed.

"Early stage standardisation (ESS)," proposed by the ISO/IEC Advisory Board on Technological Trends (ABTT), is one solution for this problem. Following the ESS system, new standards to evaluate properties of engineering ceramics could be given prior to the practical usage of them. Another advantage of this ESS system is that the standards enacted by the ESS system would introduce an additional function to develop engineering ceramics themselves. The conventional standards just focus on the fair trade of products and the rationalization of manufacturing, and therefore, the items of standards directly relate to the practical performance of materials. In other words, the purpose of this type of standards is not to clarify the essential characterization of the materials. On the other hand, new type standards based on the ESS system could pay attention to basic physical characterization and manufacturing processes of materials. Consequently, the standards enacted by the ESS system have an essential function to accelerate the research and development as well as the usage of engineering ceramics [19].

The approach to developing standards for engineering ceramics is changing from the simple arrangement of past, completed research activities to the early

stage standardisation with new research and development. Active research and development is required to promote the early stage standardisation for engineering ceramics.

The standards in flexure testing are intended for use by manufacturers and purchasers of engineering ceramics for material development, quality control, characterization and design data acquisition purposes. To develop suitable standards for flexure test method which can be easily performed and possesses accurate and consistent results, firstly the standardisation activities and the characteristics of engineering ceramics shall be reviewed. Secondly, the existing flexure test methods and the factors influencing the strength shall be studied. Thirdly, a series of step-by-step experiments must be carried out in which the effects of testing parameters on the accuracy of flexure testing are investigated. Finally, based on these results, the important characteristic features governing flexure testing are determined and appropriate flexure test standards are proposed.

The following items shall be considered as the requirements for the standard when selecting the test method:

1. theoretical validity of the test method
2. reproducibility of the measured value
3. convenience

The strength level determined by the test is calculated on the basis of linear elastic bending of a thin disc on the assumption that the material being tested is elastically homogeneous and isotropic, and shows linear (Hookean) stress-strain behaviour. Valid use of the linear elastic equation to determine centre stress stipulates that the deflection of the plate at its centre shall not exceed one half of the specimen thickness.

Ceramic materials are considered to be brittle or perfectly elastic, that is, fracture normally occurs at the surface under a tensile stress caused by flexure. The stress is termed the modulus of rupture. The modulus of rupture obtained from a strength test is influenced by a large number of factors associated with the microstructure of the material, the surface finishing procedure applied in preparation of the test pieces, the size and shape of the test piece, the mechanical

function of the testing apparatus, the rate of load application and the relative humidity of the ambient atmosphere.

The effects of time-dependent phenomena, such as stress corrosion or slow crack growth on strength tests conducted at ambient temperature, can be meaningful even for the relatively short times involved during testing. Such influences must be considered if flexure tests are to be used to generate design data.

This methodology places closely defined restrictions on the size and shape of the test piece and on the function of the test apparatus in order to minimize the errors that can arise as a consequence of the test method.

All other test factors are required to be stated in the test report in order to allow intercomparison of material behaviours. The extrapolation of flexure strength data to other geometries of stressing, to other rates of stressing or to other environments should be avoided.

With this methodology the thesis investigates the faults and omissions of existing work and judges today's requirements thereby constructing a framework with which today's and future standards in flexure testing of engineering ceramics can be based.

5.3 The proposed biaxial flexure test standard

This draft standard has been prepared and is presented in this thesis, as a result of the theoretical analysis and experimental investigation of three major techniques of the biaxial flexure tests for engineering ceramics, and shall be submitted to the standards organization (British Standards Institution, European Committee for Standardisation and International Organization for Standardisation) for the wide circulation, modification, approval and publication.

This draft standard consists of three Parts:

Part 1: The ring-on-ring test method

Part 2: The ball-on-ring test method

Part 3: The 4-Ball test method

5.3.1 The ring-on-ring test method

This part of draft standard with the provisional title of "Engineering ceramics–Determination of biaxial flexural strength at room temperature–Part 1: The ring-on-ring test method" shall contain the following content:

1. Scope

This part of the draft standard describes the testing method for determining the biaxial flexure strength (modulus of rupture) of engineering ceramics to be used as high strength materials of machine parts, structural materials etc. at room temperature.

This test method involves supporting a circular plate on a ring and loading with a small concentric ring. This test method is applicable to disc specimens in the as-fired condition or to test pieces prepared to have a certain thickness or surface finish.

This part of the draft standard does not purport to address all of the safety problems associated with its use. It is the responsibility of the user of this standard to consult and establish appropriate safety and health practices, and to determine the applicability of regulatory limitations prior to use.

2. Normative references

This part of the draft standard incorporates dated or undated references and provisions from other publications. These normative references are cited at the appropriate places in the text and the publications are listed hereafter. For dated references, subsequent amendments to or revisions of any of these publications apply to this standard only when incorporated in it by amendment or revision. For undated references, the latest edition of the publication referred to applies.

These normative references are:

ENV 623-4 Advanced technical ceramics–Monolithic ceramics–General and textural properties

Part 4: Surface roughness

DD ENN 843-2 Advanced technical ceramics–Monolithic ceramics–Mechanical properties at room temperature

Part 2: Determination of elastic moduli

EN 10002-2 Tensile testing of metallic materials

Part 2: Verification of the force measuring system of the tensile testing machine

ISO 3611 Micrometer callipers for external measurement

ISO 4677-1 Atmospheres for conditioning and testing–Determination of relative humidity

Part 1: Aspirated psychrometer method

ISO 4677-2 Atmospheres for conditioning and testing–Determination of relative humidity

Part 2: Whirling psychrometer method

3. Definitions

For the purposes of this part of the draft standard, the following definitions apply:

(1) biaxial flexure strength:

The maximum stress in a biaxial mode of flexure that a specimen develops at rupture. This stress will normally be the calculated maximum radial tensile stress at the centre of the convex surface. This mode of flexure is a cupping of the circular plate caused by central loading and supporting near the rim.

(2) ring-on-ring test:

A means of bending a thin circular disc test piece whereby the test piece is supported on a ring near its periphery, and is loaded with a small concentric ring. Configuration of the test is shown in Fig. 5.2.

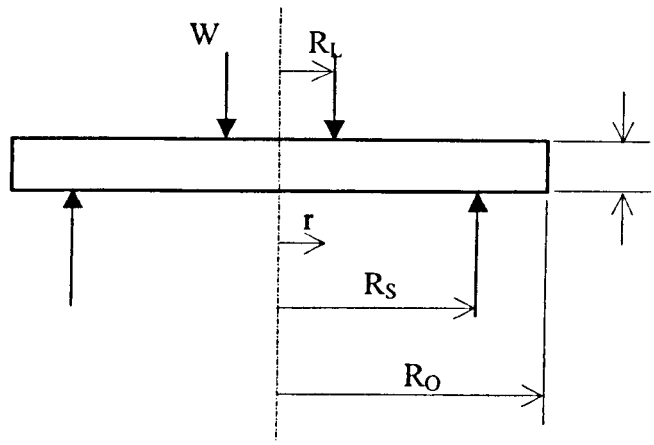


Fig. 5.2 The ring-on-ring test configuration

4. Apparatus

For the purposes of this part of the draft standard, the following apparatuses apply:

(1) test jig:

A recommended test jig is shown in Fig. 5.3. The test piece is supported on a ring near its periphery and is loaded with a small concentric ring. The jig was designed to have a 40 mm outer ring diameter and a 10 mm inner ring diameter. The rings were made from die steel (hardened and tempered) and top-surface of the ring is radiused to 5 mm. A high carbon chrome alloy steel ball with a 10 mm diameter was used to apply the load centrally to the loading ring. The load and support act through 2 mm radius toroids to minimize friction. The test jig shall be designed to have no eccentricity of loading and possess the self-aligning of the planes.

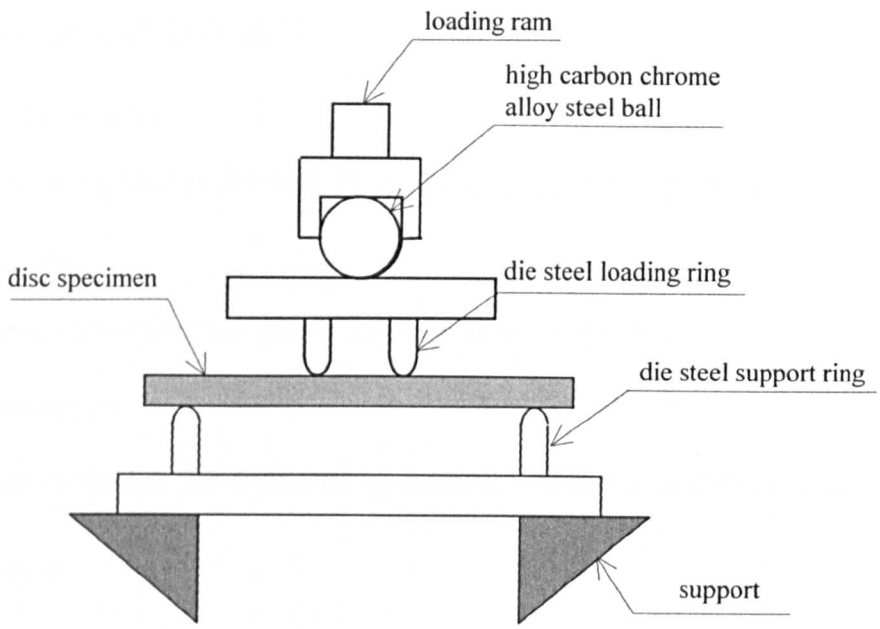


Fig. 5.3 Schematic of the ring-on-ring test jig

(2) test machine:

A suitable universal material testing machine capable of applying a force to the loading ring to stress the test piece shall be used. The machine shall also be capable of applying the force at a constant loading or displacement rate. The test machine shall be equipped for recording the peak load applied to the test piece. The accuracy of the test machine shall be in accordance with EN 10002-2, Grade 1 (accuracy 1% of indicated load) and to ensure that the force calibration on the test machine has been checked in accordance with EN 10002-2.

(3) measuring devices:

A micrometer in accordance with ISO 3611, capable of recording to 0.01 mm and accurate to this level is used to measure the thickness and diameter of test piece.

(4) drying oven:

A drying oven is used for drying the test piece after preparation at a temperature of 150 to 200 °C.

(5) humidity measuring device:

A device for measuring relative humidity to an accuracy of ±2%, e.g. those in accordance with ISO 4677.

(6) surface grinder:

A surface grinder for test piece preparation as specified in clause 6(3).

(7) desiccator:

A desiccator for test piece storage prior to testing.

(8) thermometer:

A thermometer for measuring ambient–test room temperature.

5. Test pieces

For the purposes of this part of draft standard, the following information concerning test piece apply:

(1) general:

The test pieces shall be selected and prepared according to agreement between the parties. They may either be specially processed to, or close to, the final required dimensions specified below, or may be machined from larger blocks or components. On occasion it may be desirable to test specimen geometries that fall outside the scope of this method. In such a case it is still advisable to follow the guidelines given in this standard concerning jig function to minimize errors of measurement.

(2) dimensions and tolerances:

The test pieces shall be formed or cut to size by suitable methods (see 5(3)) with care being taken that as-fired test surfaces shall be protected during processing. The test pieces shall be 43.00 ±2.15 mm in diameter. Thickness of as-fired test pieces shall not be specified except as to the minimum thickness required to limit the deflection of the test piece centre to one half the test piece thickness at fracture. Edge finish also is not specified.

(3) surface finish:

The test piece may be tested in the as-fired condition without further

surface preparation provided that they have dimensions within the tolerance given in clause 5(2) above. The test pieces may be prepared by machining in any relevant manner. If ground, rather than as-fired, surfaces are desired, the test pieces shall be ground to thickness using a 180 mesh or finer diamond grit surface grinding wheel with a wheel-surface to test piece-surface relative feed rate not exceeding 30 m/s. The stock removal rate shall not exceed 0.03 mm downfeed and 0.3 mm crossfeed per pass to the last 0.03 mm. The final 0.03 mm shall be removed with a maximum downfeed of 0.005 mm and with a maximum crossfeed of 0.12 mm per pass. Equal stock shall be removed from both the test surfaces tension (support) and compression (loaded). The flow chart of the procedures is shown in Fig. 5.4. These surfaces shall be parallel to within 0.003 mm after grinding. The surface roughness R_{max} measured using a profilometer with a stylus tip radius of less than 5 :m shall be less than 2 :m (ignoring obvious pores, see EN 623-4).

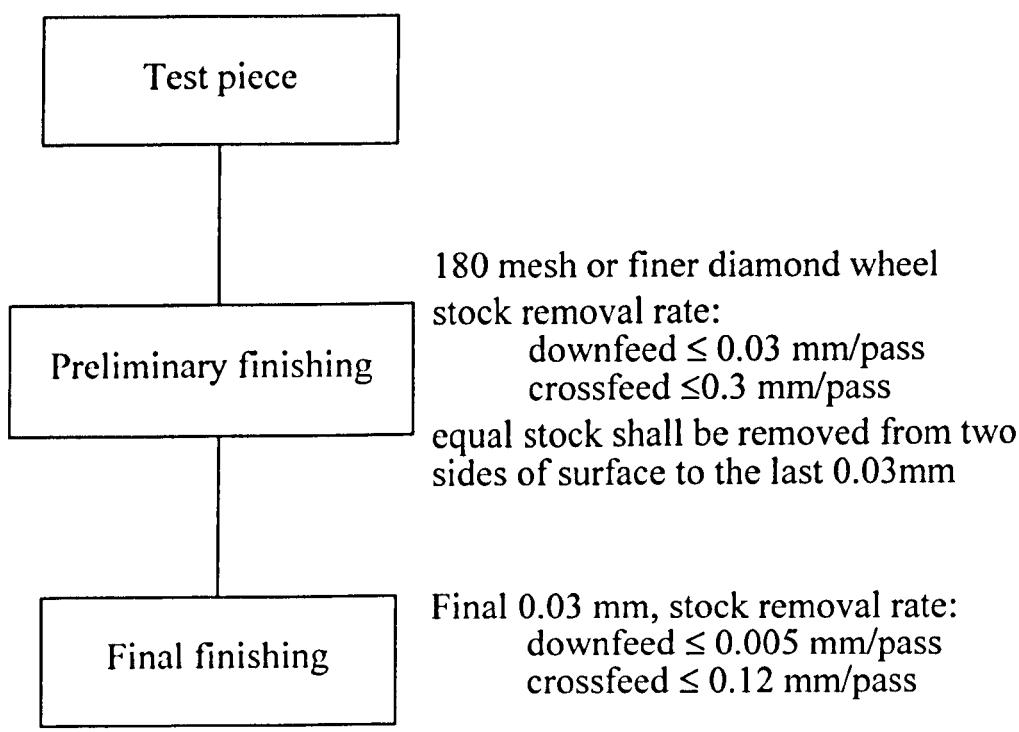


Fig. 5.4 Flow chart of the procedures for surface finish

(4) test piece conditioning:

The test pieces shall be cleansed in detergent and hot water (50 to 60 °C) followed by a hot water (50 to 60 °C) rinse. The test pieces shall be dried in an oven at 150 to 200 °C for at least 1 hour and cooled to room temperature in a desiccator.

(5) test piece defects:

No cracks, porous areas, or scratches shall be visible within the support circle of the tension surface of the test piece.

(6) number of test pieces:

For material development, characterization or quality control, the minimum number of test pieces shall be 10. For statistical evaluation of strength data (e.g. Weibull parameters) the minimum number shall be 30. Note that Weibull parameters may be seriously in error if the number of nominally identical test pieces is less than 30. The uncertainty in the parameters is sufficient to render comparisons between materials meaningless.

(7) precautions:

The prepared test pieces should be handled with care to avoid the introduction of damage subsequent to the machining process. Test pieces should be kept separate at all times, and should be individually wrapped for transport.

6. Test procedures

The test shall be subject to the following procedures.

(1) Record the ambient temperature of the test environment and the relative humidity (%RH). The temperature shall be between 15 °C and 30 °C, and shall not vary by more than 3 °C, nor the relative humidity by more than 10%, during the course of the test series.

(2) Choose a recording range for force on the testing machine (when necessary), such that the expected average force at fracture is near the centre of the range.

(3) Remove the test piece from the desiccator. Measure and record the

diameter and thickness of each test piece at three locations about 60° apart, using the micrometer (see 4(3)).

(4) Mark the tensile face of the test piece with a pencil to identify it as such.

(5) Ensure that the test jig is cleansed of fracture debris from previous tests.

(6) Position each test piece in turn in the test jig, marking the position of the points of load application and ensuring that the test piece is positioned centrally across the support ring and that the test piece is centralized under the loading axis.

(7) Place a protective screen around the test piece to trap the fractured fragments for safety reasons and for subsequent examination.

(8) Select a rate of load application. A machine displacement rate of typically 0.5 mm/min is a convenient starting point for most testing machines in cases where the expected strength of the material is 200 to 400 MPa. For materials which are much weaker or much stronger than this, the displacement rate may have to be respectively decreased or increased by an appropriate factor.

(9) Apply the test force at the chosen rate and record the peak load supported by the test piece at the instant of failure. Record the time to failure.

(10) Retrieve and identify the fracture fragments for later examination.

(11) Even if the test piece has failed outside the uniformly stressed zone produced by the loading ring, the result shall not be ignored, and shall be included in the report of the test series and in the calculation of nominal mean strength.

(12) If the test pieces are in the as-fired condition, remeasure the thickness of the test piece at the fracture position.

(13) Repeated the procedure for each test piece.

7. Calculations

Calculate the biaxial flexure strength (MOR) for each test piece from the following equation:

$$\sigma_f = (3W_f / 2\pi t^2)[(1+\nu) \ln (R_S / R_L)$$

$$+ (1-\nu)(R_S^2 - R_L^2)/2R_o^2] \qquad (5.1)$$

where

σ_f = the fracture stress, expressed as MPa

W_f = the fracture load, expressed as Newtons

t = thickness of the test piece (disc plate), expressed in mm

ν = Poisson's ratio of the test piece

R_S = radius of the support ring, expressed in mm

R_L = radius of the load ring, expressed in mm

R_o = radius of the test ring, expressed in mm

Calculate the mean value of biaxial flexure strength for the test lot from the following equation:

$$\overline{\sigma_f} = \frac{\sum_1^N \sigma_f}{N} \qquad (5.2)$$

where $\overline{\sigma_f}$ = mean fracture stress, expressed as Mpa

N = number of test pieces

Calculate the standard deviation (s) for the test lot from the following equation:

$$s = \sqrt{\frac{\sum_1^N (\sigma_f - \overline{\sigma_f})}{N-1}} \qquad (5.3)$$

Calculate the coefficient of variation (C.O.V.) for the test lot from the following equation:

$$C.O.V\ \% = 100\ s / \overline{\sigma_f} \qquad (5.4)$$

8. Test report

The test report shall contain the following information:

(1) name and address of the testing establishment;

(2) test date, unique identification of the report and of each page, customer name and address, and signatory;

(3) a reference to this standard;

(4) description of the test material (material type, manufacturing code, batch number, date of receipt, the values of Young's modulus and Poisson's ratio);

(5) method of production of test pieces from supplied material, if appropriate;

(6) exact method of test piece surface preparation, including all stages of machining;

(7) average ambient test temperature and average relative humidity during the tests;

(8) strain rate or crosshead rate;

(9) the average time to failure of the test pieces, expressed in seconds;

(10) the number of test pieces tested;

(11) individual nominal strength values for each test piece tested, expressed in MPa to three significant figures;

(12) unless otherwise agreed, the mean nominal strength and the standard deviation;

(13) the coefficient of variation for the test lot;

(14) comments about the test or the test results, details of any necessary deviations from this standard, and any observations of the nature of the fracture, and the positions and identifications of the fracture origins.

5.3.2 The ball-on-ring test method

This part of the draft standard with the provisional title of "Engineering ceramics–Determination of biaxial flexural strength at room temperature–Part 2: The ball-on-ring test method" shall contain the following content:

1. Scope

The content is the same as the description in Section 5.3.1, except the following sentence needed to be changed.

"This test method involves supporting a disc plate on a ring and centrally loading with a ball."

2. Normative references

The content is the same as the description in Section 5.3.1.

3. Definitions

The content is the same as the description in Section 5.3.1, except the following paragraph needed to be changed.

"(2) ball-on-ring test

A means of bending a thin circular disc test piece whereby the test piece is supported on a ring near its periphery, and is centrally loaded with a ball. Configuration of the test is shown in Fig. 5.5.

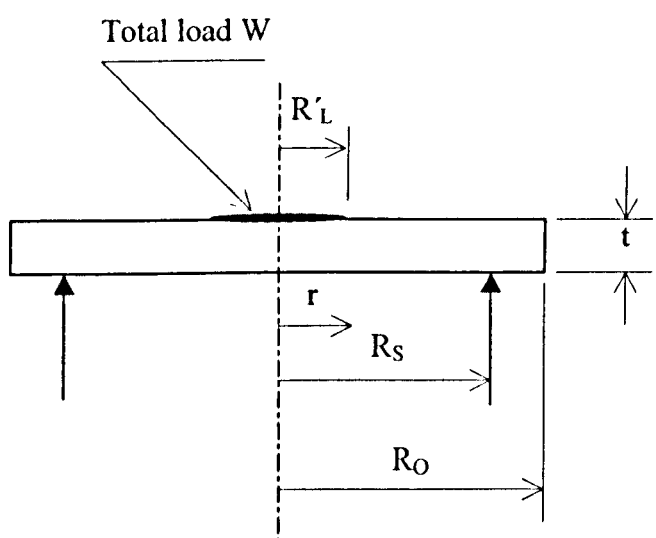


Fig. 5.5 The ball-on-ring test configuration"

4. Apparatus

The content is the same as the description in Section 5.3.1, except the following paragraph needed to be changed.

"(1) test jig:

A recommended test jig is shown in Fig. 5.6. The test piece is supported on a ring near its periphery, and is centrally loaded with a ball. The jig was designed to have a 30 mm supported ring diameter. The ring was made from die steel (hardened and tempered) and top-surface of the ring is radiused to 5 mm. The ball used to apply the load was made from high carbon chrome alloy steel (AISI 52100) and had a diameter of 5 mm. The load and support act through 2 mm radius toroids to minimize friction. The test jig shall be designed to have no eccentricity of loading and possess the self-aligning of the planes.

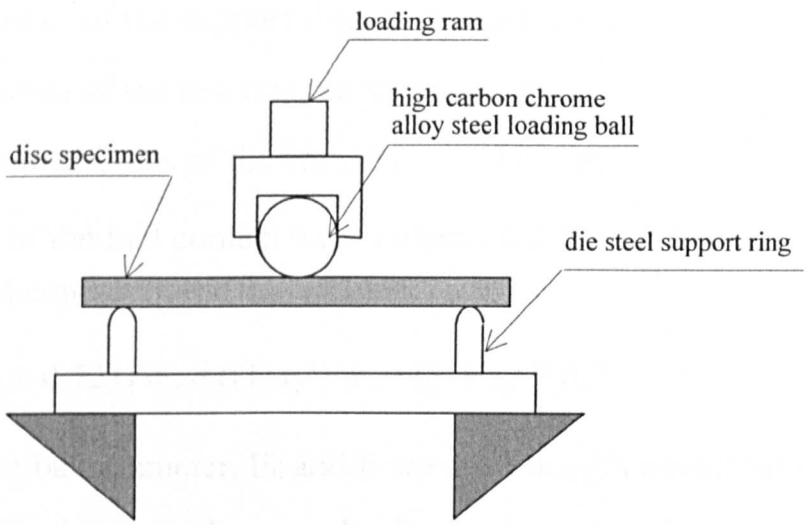


Fig. 5.6 Schematic of the ball-on-ring test jig"

5. Test pieces

The content is the same as the description in Section 5.3.1.

6. Test procedures

The content is the same as the description in Section 5.3.1.

7. Calculations

The content is the same as the description in Section 5.3.1, except the following paragraph needed to be changed.

"Calculate the biaxial flexure strength (MOR) for each test piece from the following equation:

$$\sigma_f = \frac{3W_f(1+\nu)}{4\pi t^2}\left\{1+2\ln\frac{R_S}{R_L'}+\left(\frac{1-\nu}{1+\nu}\right)\left[1-\frac{R_L'^2}{2R_S^2}\right]\frac{R_S^2}{R_O^2}\right\} \qquad (5.5)$$

Where

σ_f = the fracture stress, expressed as MPa

W_f = the fracture load, expressed as Newtons

t = thickness of the test piece (disc plate), expressed in mm

ν = Poisson's ratio of the test piece

Rs = radius of the support ring, expressed in mm

R_O = radius of the test ring, expressed in mm

R'_L =contact radius of the ball, expressed in mm

Details of the ball contact were determined from a formula given by Roark. The radius of contact of the ball (R'_L) is

$$R'_L = 0.721[W_f d((1-\nu_b^2)/E_b + (1-\nu^2)/E)]^{1/3} \qquad (5.6)$$

where d is the ball diameter, E_b and E are the Young's moduli of the ball and test piece respectively, ν_b and ν are the Poisson's ratio of the ball and test piece respectively."

8. Test report

The content is the same as the description in Section 5.3.1.

5.3.3 The 4-Ball test method

This part of the draft standard with the provisional title of "Engineering ceramics–Determination of biaxial flexural strength at room temperature–Part 3: The 4-Ball test method" shall contain the following content:

1. Scope

The content is the same as the description in Section 5.3.1, except the

following sentence needed to be changed.

"This test method involves a ball-loaded disc supported by three equi-spaced balls."

2. Normative references

The content is the same as the description in Section 5.3.1.

3. Definitions

The content is the same as the description in Section 5.3.1, except the following paragraph needed to be changed.

"(2) 4-Ball test:

A means of bending a thin circular disc test piece whereby the test piece is supported on three equi-spaced balls, and is centrally loaded with a ball. Configuration of the test is shown in Fig. 5.7.

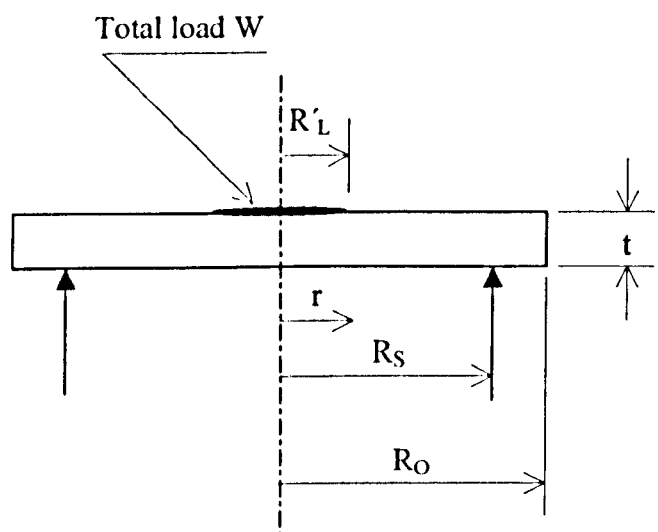


Fig. 5.7 The 4-Ball test configuration"

4. Apparatus

The content is the same as the description in Section 5.3.1, except the

following paragraph needed to be changed.

"(1) test jig:

A recommended test jig is shown in Fig. 5.8. The test piece is supported on three equi-spaced balls and is centrally loaded with a ball. The jig was designed to have a 30 mm pitch circle diameter of equi-spaced supporting balls. The four balls (the loading ball and three supporting balls) in any one 4-Ball test were identical. The balls were made from high carbon chrome alloy steel (AISI 52100) and had a diameter of 5 mm. The load and support act through 2 mm radius toroids to minimize friction. The test jig shall be designed to have no eccentricity of loading and possess the self-aligning of the planes.

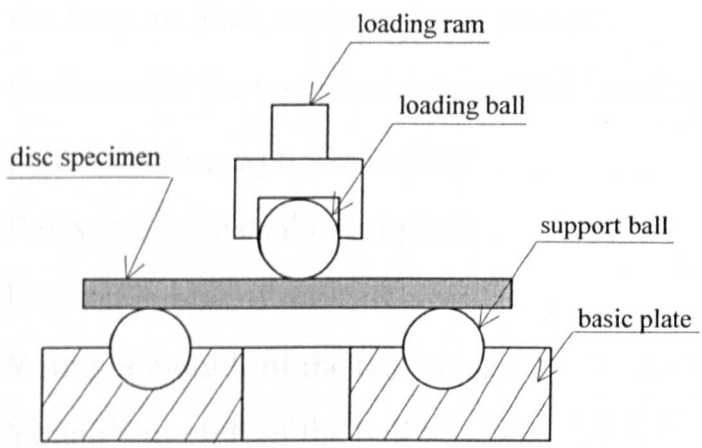


Fig. 5.8 Schematic of the 4-Ball test jig"

5. Test pieces

The content is the same as the description in Section 5.3.1.

6. Test procedures

The content is the same as the description in Section 5.3.1.

7. Calculations

The content is the same as the description in Section 5.3.1, except the

following paragraph needed to be changed.

"Calculate the biaxial flexure strength (MOR) for each test piece from the following equation:

$$\sigma_f = (3\ W_f / 2\pi t^2)\ [(1+\nu)\ln(R_S/R'_L) + (1+\nu)/2 + (1-\nu)\ (2R_S^2 - R'_L)/4R_O^2] \qquad (5.7)$$

and

$$R'_L = 0.721[W_f\ d\ ((1-\nu_b^2)/E_b + (1-\nu^2)/E)]^{1/3} \qquad (5.8)$$

where

σ_f = the fracture stress, expressed as MPa

W_f = the fracture load, expressed as Newtons

t = thickness of the test piece (disc plate), expressed in mm

d = ball diameter, expressed in mm

ν = Poisson's ratio of the test piece

ν_b = Poisson's ratio of the ball

E = Young's moduli of the test piece

E_b = Young's moduli of the ball

Ro = radius of the test piece, expressed in mm

Rs = radius of the pitch circle, expressed in mm

R'_L = contact radius of the loading ball given by Roark, expressed in mm"

8. Test report

The content is the same as the description in Section 5.3.1.

5.4 Discussion

The draft standard presented in this thesis is intended for use by manufacturers and purchasers of engineering ceramics to be used as high strength materials of machine parts, structural materials etc. for material

development, quality control, characterization and design data generation purposes.

This draft standard covers the three major testing methods for determining the biaxial flexure strength (modulus of rupture) of engineering ceramics at room temperature. The ring-on-ring, ball-on-ring, and 4-Ball test methods are the standard. The determination of flexural strength at high temperatures or very low temperatures needs more consideration in test jig design and, therefore, is not covered in this draft standard.

It is recognized that the flexural strength of a group of test specimens is influenced by several parameters associated with test procedures, such factors include the loading rate, specimen size, specimen preparation, and test fixtures. To develop an easily performed, accurate and consistent biaxial flexure testing method and thus, results, the theoretical analysis and experimental investigation of the effects of testing parameters on the flexural strength were carried out and the flexure test draft standard was prepared.

The following are the main points considered during the preparation of this draft standard:

1. The ring-on-ring, ball-on-ring, and 4-Ball test fixtures were all adopted as standard.

The fixtures should be chosen to provide a balance between practical configurations and resulting errors. For most ceramics, strength depends on the effective stressed area or volume because of the statistical distribution of strength-controlling flaws. The ring-on-ring loading fixture was constructed to provide a biaxial-tension-strength test in which the effective stressed area or volume of the specimen is comparable to the conventional uniaxial-flexture tests, such as the four-point beam bend tests. The ring-on-ring loading fixture is preferred when the determination of strength for design purposes is desired, because the circular area of the loading ring is uniformly loaded and an exact analytical stress solution is available for it.

Plate bending by the ball-on-ring method is an attractive technique, since the test is not critically affected by poor specimen tolerance. The ball-on-ring

loading fixture has advantages in precise knowledge of the stresses produced in the specimen, simple test fixtures and specimen geometry, and minimum requirements for alignment. The ball-on-ring loading fixture is preferred when investing material or process development, or when attempting to pinpoint fracture origin location.

The 4-Ball test is simple to perform and has an advantage in that support of the specimen on three balls allows the use of a slightly warped or irregular specimen. The 4-Ball loading fixture has much in its favour since contact with all four balls is assured, however there is no known exact analytical stress solution for such a case. The 4-Ball loading fixture is preferred when quality control is desired.

Each of these three systems is suited for a particular application and each has different advantages and disadvantages. In this draft standard, the disc specimen can be tested in any of the ring-on-ring, ball-on-ring and 4-Ball test fixtures.

2. The fixture size:

In the experimental investigation of the effects of testing parameters on the strengths, it was found that the fixture size of the test jig did strongly influence either the fracture strength or the value of the coefficient of variation of test results.

In the ring-on-ring test, for the same loading rate, the larger the outer ring diameter to inner ring diameter ratio, the greater the mean fracture stress obtained. The test with an outer ring diameter of 40 mm and an inner ring diameter of 10 mm showed the smallest value of coefficient of variation. For the sake of a valid comparison to uniaxial flexure, it was desired to have a similar Weibull volume or surface area. The four-point spans of uniaxial beam bending test had been of the order of 40 mm × 20 mm, but such dimensions for the ring diameters in biaxial loading would have required very high loads. Thus, an outer ring diameter of 40 mm was used with an inner ring diameter of 10 mm in this draft standard.

In the ball-on-ring test, the load ball diameter did slightly influence the

fracture strength. The mean fracture stress obtained from the ball-on-ring test using a 5 mm load ball diameter was slightly greater than that obtained using a 10 mm ball diameter. However, the coefficient of the variation of test results obtained from a 5 mm ball diameter was smaller than that obtained from a 10 mm ball diameter. For the sake of a valid comparison to the three-point uniaxial beam bending test as descried in JIS R1601, it was desired to have a load ball with a 5 mm diameter and to have a support ring with a 30 mm diameter in this draft standard.

In the 4-Ball test, the pitch circle diameter of the test jig did not strongly influence the fracture strength. The mean fracture stress obtained from the 4-Ball test using a 30 mm pitch circle diameter did not differ significantly from that obtained using a 40 mm pitch circle diameter. However, the coefficient of the variation of test results obtained from a 30 mm pitch circle diameter was smaller than that obtained from a 40 mm pitch circle diameter. On the other hand, the ball diameter did slightly influence the fracture strength. The mean fracture stress obtained using a 5 mm ball diameter was slightly greater than that obtained using a 10 mm ball diameter. The coefficient of variation of test results obtained from a 5 mm ball diameter was smaller than that obtained from a 10 mm ball diameter. In this draft standard, the jig was designed to have a 30 mm pitch circle diameter and the ball was made to have a diameter of 5 mm.

3. The loading rate:

The rate of loading can have an effect on flexural strength as the result of stress corrosion mechanisms, particularly at low strain rates. In general, the slower the rate of loading, the greater the opportunity for stress corrosion phenomena to weaken the specimen. Thus, a fast loading rate are usually used in strength tests to minimize time dependent phenomena. Times to failure for typical ceramics will range from 3 to 30 seconds.

Selection of the loading rate may have to be determined by experiment, depending on the elastic compliance of the test machine, the stiffness of the test jig and the elastic properties of the test specimens. An experimental investigation of the loading rate dependence of fracture strength in 4-Ball testing was conducted in this work. It was found that the loading rate did strongly

influence the fracture strength. For the same pitch circle diameter and same ball diameter, the slower the speed of loading, the smaller the mean fracture stress obtained. It was also found that the loading rate of 0.5 mm/min has the smallest value of coefficient of variation.

A machine displacement rate of typically 0.5 mm/min is a convenient starting point for most testing machines in cases where the expected strength of the material is 200 to 400 MPa. For materials which are much weaker or much stronger than this, the displacement rate may have to be respectively decreased or increased by an appropriate factor.

4. Specimen size:

The strength of a ceramic can be dependent upon test specimen size. In general, the larger the specimen, the weaker it is likely to be. Such size influence can be analyzed via statistical theories of strength.

In the interests of permitting greater compatibility of data, specific specimen will be required by the standard. In this draft standard, one specimen size, 43.00 ±2.15 mm in diameter, was specified, which is comparable to the conventional 3mm ×4mm ×45mm uniaxial flexure test specimen size and could be tested in any of the ring-on-ring, ball-on-ring and 4-Ball test. Thickness of as-fired test specimens shall not be specified except as to the minimum thickness required to limit the deflection of the test specimen centre to one half of the test specimen thickness at fracture.

5. Specimen surface preparation:

The surface preparation of test specimens can have a pronounced effect upon flexural strength due to the introduction of machining flaws which can be strength limiting. Machining damage imposed during specimen preparation can be either a random interfering factor, or an inherent part of the strength characteristic to be measured. Surface preparation can also lead to surface residual stresses. It is recognized that the final machining steps may or may not negate machining damage introduced during the early course or intermediate machining. Universal optimum or standardised methods of surface preparation do not exist. Nevertheless, some minimum requirements will be specified in

the standard. In this draft standard, the test piece may be tested in the as-fired condition without further surface preparation provided that they have dimensions within the tolerance given in the standard. If machining is required, this draft standard gives a prescribed two-step progressively finer process.

6. Sample size:

The choice of sample size depends on many factors including the cost and timing of testing and the degree of conservation which is acceptable, but erroneous judgements may be made and unacceptable designs pursued if the sample sizes are too small. Statistical analysis shows that wide variances in mean strengths and Weibull parameters are normal for samples with as few as 10 specimens. The number of specimens required for the flexure testing has been specified. In this draft standard, a minimum of 10 specimens shall be required for the purpose of material development, characterization or quality control and a minimum of 30 specimens shall be necessary for statistical evaluation of strength data (e.g. Weibull parameters).

The flexure strength of a ceramic is not a deterministic quantity, but will vary from one specimen to another. There will be an inherent statistical scatter in the results for finite sample sizes. The three major biaxial test methods prescribed in this draft standard has been devised so that the precision is very high and the bias very low compared to the inherent variability of strength of the material. However, the uncertainty of measurement in flexure testing always exists and needs to be estimated.

CHAPTER 6
Estimation of Uncertainty of Measurement in Flexure Testing

6.1 Introduction

A measurement is a set of operations having the objective of determining the value of the measurand. The measurand is the particular quantity to be measured. A measurement therefore begins with an appropriate specification of the measurand, the method of measurement, and the measurement procedure [87].

Measurements are made to obtain data to enable decisions to be made and actions to be taken. Electricity supply authorities measure the amount of power supplied to their clients in order to have a reasonable basis on which to calculate period charges. Measurements of time are made by the community in order to regulate the smooth operation of commerce, business and government. Manufacturers need to make measurements to determine whether components have been made within tolerance and can be properly combined with other components to make assemblies. Measurements of flexural strength of engineering ceramics are made to assume primary structural functions at high stress levels, even under dynamic loading.

Measurement activities are often complex procedures containing several steps. Each step of the procedure includes different components and elements and each component is combined with factors and phenomena which influence the outcome of the measurement procedure as a whole, for example temperature and humidity or stray electrical fields can introduce variations into results of measurements. These variations in different elements of a measurement procedure depend on the fact that there are a number of factors which influence the testing program. It is often not possible to hold these influencing factors completely constant. The combined result of these influences gives rise to an error in the measurement result.

In many industrial and commercial applications, as well as in the areas of health and safety, it is often necessary to provide a statement with a high level of confidence that the measurement result will fall within certain values.

The concept of uncertainty as a quantifiable attribute is relatively new in the history of measurement, although error and error analysis have long been a part of the practice of measurement science or metrology. Measurement standards of mass and length dating back 5000 years have been unearthed. It is not known when the first debate over measurement accuracy or the assessment of measurement uncertainty happened. In the sixteenth century, Johannes Kepler (1571-1630) was forced to reject years of work on the calculation of planetary orbits when he found that the discrepancy between his calculated positions was much greater than the uncertainties of observations made by Tycho Brahe (1546-1601). Kepler eventually found orbit's true shape to be elliptical. Isaac Newton (1642-1727) was later able to build on Kepler's work to derive his own laws of motion. Thus, much of the theory behind modern engineering and physics developed because of some simple but well-calibrated instruments and measurements with a known uncertainty [88].

About two hundred years ago, as the Industrial Revolution gathered speed, in Britain, France and the United States, more prosaic needs for accurate measurement were developed. The development of machine tools, steam engines and the mass production of goods, which needed to have interchangeable components for maintenance and repair, all exerted strong pressure to have good control over manufacturing tolerance and measurement uncertainty.

No manufactured part is ever made exactly to size, and no instrument or equipment is ever made exactly to specification. There is always a manufacturing tolerance, and the specified quantities must always be measured with some uncertainty. Only by correctly and consistently calculating these quantities can functional and reliable objects be made. The most successful companies to emerge from the industrial revolution were those that paid attention to their measurement practices.

It is now widely recognized that when all of the known or suspected

components of error have been evaluated and the appropriate corrections have been applied, there still remains an uncertainty about the correctness of the stated result, that is, a doubt about how well the result of the measurement represents the value of the quantity being measured.

A knowledge of uncertainty of measurement is necessary to decide on whether to accept or reject items being measured for conformance to specification, to decide on the number of significant digits in engineering constants and instrument corrections and to decide on the appropriateness of a measurement system.

Uncertainty analysis is also a powerful tool in determining which part of a measurement system is the most appropriate to improve if better measurements are required. A tool, such as a spreadsheet, can be used to analyse the effect of better temperature control, a better volt meter, more measurements and so forth [89].

The general approach that has been used in the past consisted of minimising errors in measurements by calibrating the measuring instrument, applying every correction that was thought necessary and then repeating the measurement a number of times. The dispersal of the repeated measurements was usually taken as the sole indicator of measurement error.

Improved instrumentation with excellent resolution has meant that the residual errors due to imperfect calibration, imperfect corrections and the like, were greater than the resolution and therefore warranted consideration. Various schemes were developed with the variations of repeated readings being regarded as arising from random errors and the other components as being systematic errors.

The problem that then arose was how to combine these two types of errors. Eventually, it was recognised that both could be described by one type of parameter, a variance, but even the method of combination promoted by the British National Physical Laboratory (NPL) through their British Calibration Service (BCS) sometimes gave illogical results. A variation in the standard method was devised which worked well, but the NPL approach was not universally accepted and although it appeared simple to apply in practice it

required significant skill. It was, nevertheless, an advance on the approach of estimating worst case limits for all uncertainty components and then simply adding them up to get a worst case (or probable) total uncertainty [88].

Just as the nearly universal use of the International System of Units (SI) has brought coherence to all scientific and technological measurements, a worldwide consensus on the evaluation and expression of uncertainty in measurement would permit the significance of a vast spectrum of measurement results in science, engineering, commerce, industry, and regulation to be readily understood and properly interpreted. In this era of the global marketplace, it is imperative that the method for evaluating and expressing uncertainty be uniform throughout the world so that measurements performed in different countries can be easily compared.

To try to achieve an international consensus of the expression of uncertainty in measurement, the International Committee for Weights and Measures (CIPM) requested the International Bureau of Weights and Measures (BIPM) in 1978 to address the issue of expression of uncertainty in measurement.

In 1980, BIPM published their recommendations (Recommendation INC-1 (1980), The Expression of Experimental Uncertainties). This recommendation was adopted by CIPM in 1981 and reaffirmed in 1986.

The task to develop a more detailed guidance document based on these recommendations was later referred by CIPM to the International Standards Organisation (ISO), because ISO would be able to better reflect the needs arising from the broad interests of industry and commerce. In 1993, the ISO Guide to the expression of uncertainty in measurement was published.

The ISO Guide presents a unified approach to uncertainty assessment, on which has a good mathematical basis, and allows for the application of experience and knowledge when appropriate for estimation. The results are in good agreement with older well-accepted schemes. The guide is universal in that it can be applied to any kind of measurement. It is internally consistent and the results can be transferred into calculations performed by other parities.

In this chapter, the methodology for the estimation of uncertainty in measurement based on the ISO Guide is described. Some common categories of uncertainty sources are presented. The procedure and results of the estimations of uncertainty in measurement for the proposed biaxial flexure test standard are presented and discussed.

6.2 Methodology

From the time the first measurement was made, the desire to determine the accuracy of measurements in a meaningful way has been a topic for lively debate. Around 1970, it was widely recognised that an uncertainty was necessary for every measured value, so that the measurement quality could be accessed for fitness of purpose. There remained two problems. Firstly, how should uncertainty be assessed. Secondly, because there were many opinions and hence methods of assessment, comparisons were difficult. Research papers involving highly accurate measurements and reports of high level intercomparisons devoted pages to explaining how the uncertainties were assessed so that they could be redone by others in alternative ways.

The ideal method for evaluating and expressing the uncertainty of the result of a measurement should be universal. The method should be applicable to all kinds of measurement and to all types of input data used in measurements.

The actual quantity used to express uncertainty should be internally consistent and transferable. It should be directly derivable from the components that contribute to it, as well as independent of how these components are grouped and of the decomposition of the components into subcomponents. It also should be possible to use directly the uncertainty evaluated for one result as a component in evaluating the uncertainty of another measurement in which the first result is used.

In many industrial and commercial applications, it is often necessary to provide an interval about the measurement result that may be expected to encompass a large fraction of the distribution of values that could reasonably be attributed to the quantity subject to measurement. Thus, the ideal method for evaluating and expressing uncertainty in measurement should be capable of

readily providing such an interval, in particular, one with a coverage probability or level of confidence that corresponds in a realistic way with that required [87].

The approach upon which the ISO Guide to the expression of uncertainty in measurement is based meets all of the requirements outlined above. This is not the case for most other methods in current use. There are two significant ideas which appear in the guide which are different from previous methods. Firstly, the concept of there being only two classes of evaluation, type A and Type B. The previous categories of random and systematic uncertainties are no longer used because they have inappropriate connotations for uncertainty. The second innovation is the use of effective degrees of freedom to allow the combined uncertainty of the measurement to have a defined confidence level which is sufficiently high for practical use, e.g. 95%.

The adoption of the ISO Guide possesses many advantages. The ISO Guide offers a universal method that is internationally recognised and suitable for evaluation and expressing the uncertainty of the result of a measurement. The method is applicable to all kinds of measurement and to all types of input data used in measurements and uncertainty calculation. Because the method is well defined, internally consistent and independent of the uncertainty components, it provides results that are transferable from one measurement to another, independently of who does the work or where it is done.

The uncertainty in measurement for the biaxial flexure test standard proposed in this thesis are estimated using the methodology in the ISO Guide to the expression of uncertainty in measurement (2nd edition, 1995). It will be summarized as follows [87].

6.2.1 Modelling the measurement

The physical measurement system is modelled by representing it in the form of an equation, a formula, a diagram or perhaps a description. The model is a formal description of the system. If the model is a valid representation of the physical system, then it is possible to investigate the behaviour of the system. The development of a model is crucial to the estimation of measurement

uncertainty.

It is necessary to consider all the effects, influences and other contributors to the uncertainty of a measurement. Listing these is a useful tool, as is forming a model of the measurement process or system. The model may be a simple linear one in which the components can be separately calculated or a complex system of equations.

In most cases a measurand Y is not measured directly, but is determined from N other quantities $X_1, X_2, \ldots X_N$ through a functional relationship f:

$$Y = f(X_1, X_2, \ldots X_N) \qquad (6.1)$$

The input quantities $X_1, X_2, \ldots X_N$ upon which the output quantity Y depends may themselves be viewed as measurands and may themselves depend on other quantities, including corrections and correction factors for systematic effects, thereby leading to a complicated functional relationship f that may never be written down explicitly. Further, f may be determined experimentally or exist only as an algorithm that must be evaluated numerically.

Thus, if data indicate that f does not model the measurement to the degree imposed by the required accuracy of the measurement result, additional input quantities must be included in f to eliminate the inadequacy. This may require introducing an input quantity to reflect the incomplete knowledge of a phenomenon that affects the measurand.

The set of input quantities $X_1, X_2, \ldots X_N$ may be categorized as:

(1) quantities whose values and uncertainties are directly determined in the current measurement. These values and uncertainties may be obtained from, for example, a single observation, repeated observations, or judgements based on experience, and may involve the determination of corrections to instrument readings and corrections for influence quantities, such as ambient temperature, barometric pressure and humidity.

(2) quantities whose values and uncertainties are brought into the measurement from external sources, such as quantities associated with calibrated measurement standards, certified reference materials and reference data

obtained from handbooks.

An estimate of the measurand Y, denoted by y, is obtained from equation (6.1) using input estimates x_1, x_2...,x_N for the values of the N quantities X_1, X_2, ...X_N. Thus the output estimate y, which is the result of measurement, is given by

$$y = f(x_1, x_2, ..., x_N) \qquad (6.2)$$

The estimated standard deviation associated with the output estimate or measurement result y, termed combined standard uncertainty and denoted by $u_c(y)$, is determined from the estimated standard deviation associated with each input estimate x_i, termed standard uncertainty and denoted by $u(x_i)$.

Each input estimate x_i and its associated standard uncertainty $u(x_i)$ are obtained from a distribution of possible values of the input quantity X_i. This probability distribution may be frequency based, that is, based on a series of observations $X_{i,k}$ of X_i, or it may be an *a priori* distribution. Type A evaluations of standard uncertainty components are founded on frequency distributions while Type B evaluations are founded on *a priori* distributions. It must be recognized that in both cases the distributions are models that are used to represent the state of our knowledge.

6.2.2 Two types of evaluation

The ISO Guide defines two types of uncertainty components: Type A and Type B, which are distinguished by their methods of evaluation.

Type A uncertainty components are evaluated by using standard statistical methods to analyse a set or sets of measurements and include those error terms previous referred to as random errors. They are characterised by an estimated variance or standard deviation, a mean value (or equivalent) and the number of degrees of freedom.

The measure which is used to characterise the dispersion of all input quantities is the variance. The variance is the expectation of the square of the centred random variable. A more convenient parameter is the standard deviation which is the square root of the variance. The variance and the

standard deviation of a set of measurements can be calculated using standard formulas. The parameters can also be easily calculated by any modern scientific calculator, although care must be taken with many calculators to ensure that the standard deviation of sets of numbers with more than three or four significant places does not fail due to unstable algorithms. The number of degrees of freedom in Type A evaluations is one less than the number of measurement.

Type B uncertainty components are those components evaluated by means other than the statistical analysis of a series of observations. They include the class of errors previously called systematic errors. Their evaluation involves finding a quantity considered to correspond to a variance, the existence of which is assumed. They are characterised by an estimated variance or standard deviation, a mean value (which may be zero), and a number of degrees of freedom.

Examples of when Type B evaluations are required include: estimation of measurement scatter when only one value is measured, i.e. no repeated measurements; readout resolution; hysteresis; finite-precision arithmetic and rounding of reported values; residuals of corrections, such as for temperature and other environmental effects; small corrections which are not applied; effects of the method of measurement; and sometimes the uncertainty of the calibration of the instrument.

In the case of Type B evaluations, the standard uncertainty is evaluated by scientific judgement based on all of the available information on the possible variability of an input quantity. The pool of information may include: previous measurement data; experience with or general knowledge of the behaviour and properties of relevant materials and instruments; manufacturer's specifications; data provided in calibration and other certificates; and uncertainties assigned to reference data taken from handbooks.

The proper use of the pool of available information for a type B evaluation of standard uncertainty calls for insight based on experience and general knowledge, and is a skill that can be learned with practice. It should be recognized that a Type B evaluation of standard uncertainty can be reliable as a

Type A evaluation, especially in a measurement situation where a Type A evaluation is based on a comparatively small number of statistically independent observations.

The purpose of the Type A and Type B classification is to indicate the two different ways of evaluating uncertainty components and is for convenience of discussion only; the classification is not meant to indicate that there is any difference in the nature of the components resulting from the two types of evaluation. Both types of evaluation are based on probability distribution, and the uncertainty components resulting from either type are quantified by variances or standard deviations.

It is important not to double-count uncertainty components. If a component of uncertainty arising from a particular effect is obtained from a Type B evaluation, it should be included as an independent component of uncertainty in the calculation of the combined standard uncertainty of the measurement result only to the extent that the effect does not contribute to the observed variability of the observations. This is because the uncertainty due to that portion of the effect that contributes to the observed variability is already included in the component of uncertainty obtained from the statistical analysis of the observations.

6.2.3 Determining combined standard uncertainty

Once all the variances, or their equivalents, have been found using the Type A and Type B evaluations, they can be combined and a combined standard deviation found.

The combined standard uncertainty is defined as the standard uncertainty of the result of a measurement when the result is obtained from the values of a number of other quantities, equal to the positive square root of a sum of terms, the terms being the variances (and covariances) of these quantities weighted according to how the measurement result varies with changes to these quantities.

Generally input quantities are considered independent of each other, but sometimes two or more of them can depend strongly on each other. In the case where all input quantities are independent, the standard uncertainty of the result

of the measurement is obtained by appropriately combining the standard uncertainties of the input estimates.

The combined standard uncertainty $u_c(y)$ is the positive square root of the combined variance $u_c^2(y)$, which is given by

$$u_c^2(y) = \sum_{i=1}^{N} \left[\frac{\partial f}{\partial x_i} \right]^2 u^2(x_i) \qquad (6.3)$$

Where f is the function given in equation (6.1). Each $u(x_i)$ is a standard uncertainty evaluated by using the Type A or Type B evaluation. The combined standard uncertainty is an estimated standard deviation and characterises the dispersion of the values that could reasonably be attributed to the measurand.

Equation (6.3) is valid only if the input quantities are independent or uncorrelated. If some of the input quantities are significantly correlated, the correlations must be taken into account.

When the input quantities are correlated, the appropriate expression for the combined variance $u_c^2(y)$ associated with the result of a measurement is

$$u_c^2(y) = \sum_{i=1}^{N} \sum_{j=1}^{N} \frac{\partial f}{\partial x_i} \frac{\partial f}{\partial x_j} u(x_i, x_j) \qquad (6.4)$$

Where x_i and x_j are the estimates of X_i and X_j and $u(x_i, x_j) = u(x_j, x_i)$ is the estimated covariance associated with x_i and x_j.

There may be significant correlation between two input quantities if the same measuring instrument, physical measurement standard, or reference datum having a significant standard uncertainty is used in their determination. For example, if a certain thermometer is used to determine a temperature correction required in the estimation of the value of input quantity X_i, and the same thermometer is used to determine a similar temperature correction required in the estimation of input quantity X_j, the two input quantities could be significantly correlated. However, if X_i and X_j in this example are redefined to

be the uncorrected quantities and the quantities that define the calibration curve for the thermometer are included as additional input quantities with independent standard uncertainties, the correlation between X_i and X_j is removed.

Correlations between input quantities cannot be ignored if present and significant. The associated covariances should be evaluated experimentally if feasible by varying the correlated input quantities, or by using the pool of available information on the correlated variability of the quantities in question (Type B evaluation of covariance). Insight based on experience and general knowledge is especially required when estimating the degree of correlation between input quantities arising from the effects of common influences, such as ambient temperature, barometric pressure, and humidity. Fortunately, in many cases, the effects of such influences have negligible interdependence and the affected input quantities can be assumed to be uncorrelated. However, if they cannot be assumed to be uncorrelated, the correlations themselves can be avoided if the common influences are introduced as additional independent input quantities.

6.2.4 Determining expanded uncertainty

Although the combined standard uncertainty $u_c(y)$ can be universally used to express the uncertainty of a measurement result, in some commercial, industrial, and regulatory applications, and when health and safety are concerned, it is often necessary to give a measure of uncertainty that defines an interval about the measurement result that may be expected to encompass a large fraction of the distribution of values that could reasonably be attributed to the measurand. The ISO Guide meets this requirement with recommendations for the calculation of an expanded uncertainty.

The expanded uncertainty is a quantity defining an interval about the result of a measurement that may be expected to encompass a large fraction of the distribution of values that could reasonably be attributed to the measurand. This is achieved by multiplying the combined uncertainty by a coverage factor that corresponds to the Student's t-factor, t, for the desired confidence level. Student's t is for the uncertainty of mean values. It is necessary to also know the degrees of freedom before looking up tables of t. As all the components

have differing degrees of freedom, weighted or effective degrees of freedom, V_{eff}, is required for use with the selection of the coverage factor. The ISO Guide gives an equation which may be used to calculate the combined degrees of freedom called the effective degrees of freedom. The expanded uncertainty gives the dispersion of values which could be attributed to the measurand with a defined level of confidence or likelihood as for example, 95% or 99%.

The value of the coverage factor k is chosen on the basis of the level of confidence required of the interval. In general, k will be in the range 2 to 3. However, for special applications k may be outside this range. Extensive experience with and full knowledge of the uses to which a measurement result will be put can facilitate the selection of a proper value of k. For the establishment of the relation between the coverage factor and level of confidence, a simple approach is often adequate in measurement situations where the probability distribution is approximately normal and the effective degrees of freedom is of significant size. When this is the case, which frequently occurs in practice, one can assume that taking k=2 produces an interval having a level of confidence of approximately 95%, and that taking k=3 produces an interval having a level of confidence of approximately 99%.

6.2.5 Reporting uncertainty

Although in practice the amount of information necessary to document a measurement result and its uncertainty depends on its intended use, the basic principle of what is required remains unchanged. When reporting the result of a measurement and its uncertainty, it is preferable to provide too much information rather than too little. The ISO Guide, clause 7, gives advice on the methods of reporting the measurement result and its associated uncertainty.

When reporting the result of a measurement, and when the measure of uncertainty is the expanded uncertainty $U = ku_c(y)$, one should:

(1) give a full description of how the measurand Y is defined ;

(2) state the result of the measurement as $Y = y \pm U$ and give the units of y and U;

(3) include the relative expanded uncertainty U/|y|, |y|≠0, when appropriate.

(4) give the value of k used to obtain U;

(5) give the approximate level of confidence associated with the interval y ± U and state how it was determined;

In the detailed report that describes how the result of measurement and its uncertainty was obtained, one should:

(1) give the value of each input estimate and its standard uncertainty together with a description of how they were obtained ;

(2) give the estimated covariances or estimated correlation coefficients (preferably both) associated with all input estimates that are correlated, and the methods used to obtain them;

(3) give the degrees of freedom for the standard uncertainty of each input estimate and how it was obtained;

(4) give the functional relationship $Y=f(X_1, X_2, ..., X_N)$ and, when they are deemed useful, the partial derivatives or sensitivity coefficients $\partial f/\partial x_i$. However, any such coefficients determined experimentally should be given.

6.3 Sources of uncertainty

The total uncertainty is made up from all the factors which contribute to the dispersion of the values that could reasonably be attributed to the measurand. The sources of these contributions to the dispersion arise from all the factors associated with the measurement. The ISO Guide sets out a comprehensive summary of the possible sources of uncertainty.

The following is a listing of some common categories of uncertainty sources [88].

(1) measurement standards and references

The measurement standard or reference against which the test piece or instrument is compared or calibrated has its own value and associated

uncertainty. Generally, each standard has been compared to a higher level standard so that the uncertainty is traced up the line of calibrations. The standard would normally be associated with a calibration report which states its value and its uncertainty. Even at the very top of the pyramid, the actual physical primary realisation of a unit, such as the metre, has an uncertainty associated with it, being a measure of the reasonable dispersion of values which could be attributed to that realisation of the unit.

(2) measurement method and procedure

There is often more than one method of finding the value of any measurand. Null methods or non-contact methods should be used when the measuring instrument may have an influence on the measurand. Methods which are required taking differences of large numbers should be avoided as a small error in the large, similar magnitude, values will be greatly magnified in the value of their difference. By arranging measurements in a particular time sequence, drift can often be substantially reduced.

Some methods actually measure a slightly different measurand due to subtle changes in the definition of the measurand when the method is changed. For example, there is an apparent difference in the position of the surface of a gauge block when measured with light instead of a mechanical probe. Approximations and assumptions incorporated in the measurement method and procedure, and nonrepresentative sampling are also examples of sources of uncertainty.

(3) workpiece and measurand

The workpiece is the object that is the subject of the measurement. The measurand is a characteristic of the workpiece that is to be measured and the degree to which the measurand can be determined is dependent on the quality of the workpiece. For example, the degree to which the flatness of a gauge block can be determined depends on the quality (surface finish, flatness) of the block itself. The quality of the test object itself contributes to the measurement uncertainty. Even with the best equipment, a mediocre test item will ensure that only a mediocre uncertainty can be achieved.

(4) instruments

The process of using an instrument can change the value of the measurand. Many electrical instruments, for example, draw finite energy from the circuit they are measuring and so create circuit loading leading to systematic errors. Another example is the elastic deformation that can occur when a micrometer is used for length measurements. While both of these effects can be measured or estimated and a correction applied, some part of the induced error will remain indeterminate, thus producing a component of uncertainty for the measured value.

Instruments are not perfect and while calibration is essential to minimise errors, residual non-lineararities, zero drifts, limited discrimination, hysteresis, scale factor drifts and electrical noise, etc. give rise to uncorrectable errors which may have a significant effect on the measured value.

(5) environmental conditions

Inadequate knowledge of the effects of environmental conditions on the measurement or imperfect measurement of environmental conditions is the common source of uncertainty. Almost every measurand has the potential to be used as a thermometer, whether it is used that way or not. Optical measurements are often affected by vibration and drifts.

Electromagnetic interference is more of a problem as personal portable telephones become more common. Most new instrumentation includes electronics which may be susceptible to external electrical interference.

If the measurand has been in a different environment to which it is to be measured, then a period of acclimatisation will be required before measurement begins.

(6) personnel

The person performing the test is a significant influential quantity. Sources of uncertainty include the effect of body heat, dexterity with adjustments (such as setting indicators to zero) and bias in reading analogue instruments.

(7) other sources of uncertainty

Incomplete definition of the measurand, imperfect realisation of the definition of the measurand, uncertainties of the values of reference standards and reference materials, finite instrument resolution or discrimination threshold, inexact values of constants and other parameters obtained from external sources and used in the data-reduction algorithm, and variations in repeated observations of the measurand under apparently identical conditions are all possible sources of uncertainty in a measurement.

6.4 Estimation of uncertainty of measurement

The clause 5.4.6.2 of ISO/IEC 17025 (1st edition,1999), general requirements for the competence of testing and calibration laboratories, specifies that testing laboratories shall have and apply procedures for estimating uncertainty of measurement. In certain cases the nature of the test method may preclude rigorous, metrologically and statistically valid, calculation of uncertainty of measurement. In these cases the laboratory shall at least attempt to identify all the components of uncertainty and make a reasonable estimation, and ensure that the form of reporting of the result does not give a wrong impression of the uncertainty. In these cases where a well-recognized test method specifies limits to the values of the major sources of uncertainty of measurement and specifies the form of presentation of calculated results, the laboratory is considered to have satisfied this clause by following the test method and reporting instructions[90]. Since the uncertainty of measurement in biaxial flexure testing has never been assessed before, this thesis is therefore firstly attempted to identify the major components of uncertainty and make a reasonable estimation for the test method proposed in Section 5.3. The estimation is based on the approach to expressing uncertainty provided in the Guide to the Expression of Uncertainty in Measurement published by the ISO in 1995. The flow chart of estimation of uncertainty of measurement is shown as Fig. 6.1.

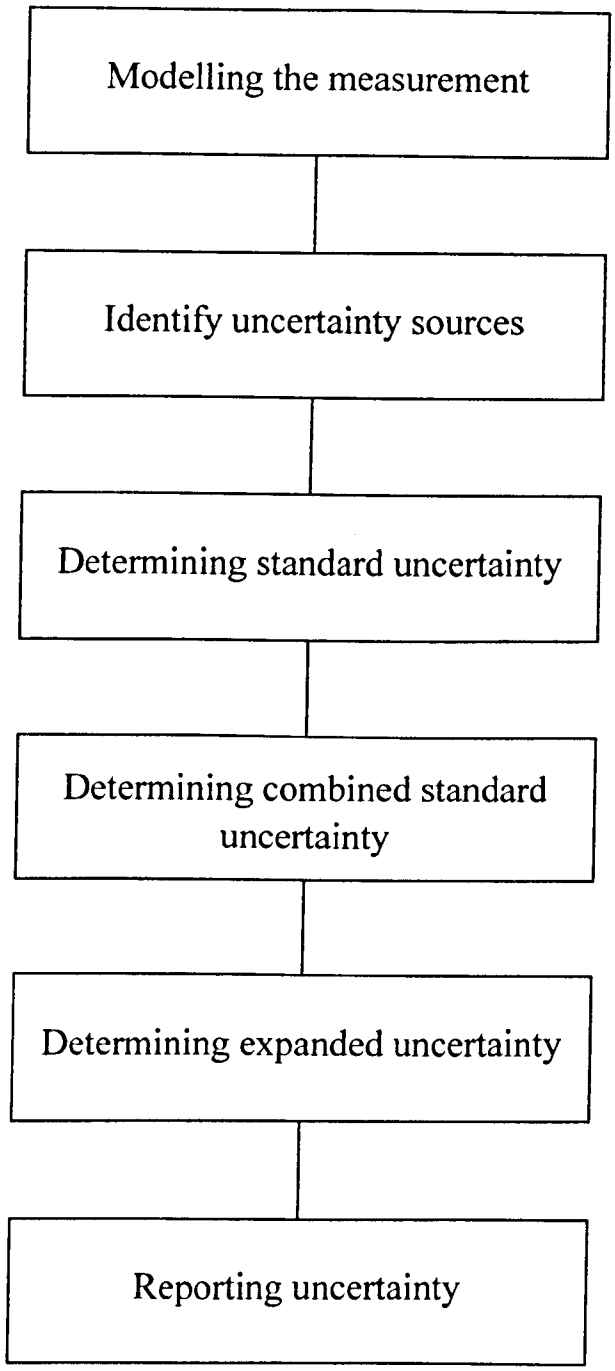


Fig. 6.1 Flow chart of estimaton of uncertainty of measurement

6.4.1 The ring-on-ring test method

The ring-on-ring test involves supporting a circular plate on a ring and loading with a small concentric ring. The tensile fracture stress determined by ring-on-ring test is obtained from the fracture load using equation (4.1) shown in Section 4.2.2. For a set of tests by the method proposed in Section 5.3.1, the mean biaxial flexural strength will be determined as follows:

$$\overline{\sigma_f} = (\sigma_1 + \sigma_2 + \text{----------} + \sigma_n)/n \qquad (6.5)$$

Since the equation (4.1) does not consider the uncertainty contributed by the random effect, it is assumed that each time the test results shall be the sum of real strength and a variance Δ.

$$\overline{\sigma_f} = [(\sigma_f + \Delta_1) + (\sigma_f + \Delta_2) + \text{----------} + (\sigma_f + \Delta_n)]/n$$

$$= \sigma_f + (\Delta_1 + \Delta_2 + \text{----------} + \Delta_n)/n \qquad (6.6)$$

Since no variance has been added, it is assumed that the variance is a normal distribution and the mean of the distribution is zero and the variance is s^2. The equation (4.1) can be rewritten as:

$$\sigma_f = (3W_f/2\pi t^2)[(1+\nu)\ln(R_S/R_L) + (1-\nu)(R_S^2 - R_L^2)/2R_o^2] + \Delta \qquad (6.7)$$

From the equation (6.7), it is known that the flexural strength is a function of the applied load, thickness of the disc plate, Poisson's ratio of the disc plate, radius of the support ring, radius of the load ring and radius of the disc plate, thus the equation (6.7) can be expressed as

$$\sigma_f = f(W,\ t,\ R_S,\ R_o,\ R_l,\ \Delta) \qquad (6.8)$$

The major components contributing to the overall uncertainty are:

u(W) : the uncertainty in measurement of the applied load
u(t) : the uncertainty in measurement of thickness of the disc plate
$u(R_s)$: the uncertainty in measurement of radius of the support ring
$u(R_o)$: the uncertainty in measurement of radius of the disc plate
$u(R_l)$: the uncertainty in measurement of radius of the load ring
$u(\Delta)$: the uncertainty contributed from random effect

It is assumed that the major components are independent of each other, the uncertainty of the mean biaxial flexural strength then can be expressed as under:

$$u_c^2(\sigma_f) = \left[\frac{\partial\sigma}{\partial W}\right]^2 u^2(W)+\left[\frac{\partial\sigma}{\partial t}\right]^2 u^2(t)+\left[\frac{\partial\sigma}{\partial R_S}\right]^2 u^2(R_S)+\left[\frac{\partial\sigma}{\partial R_O}\right]^2 u^2(R_O)$$

$$+\left[\frac{\partial\sigma}{\partial R_L}\right]^2 u^2(R_L)+\left[\frac{\partial\sigma}{\partial\Delta}\right]^2 u^2(\Delta) \qquad (6.9)$$

The applied load is given by a test machine and the test method proposed in Section 5.3.1 specifies that the accuracy of the test machine shall be 1 % of indicated load. In the test performed in this study, the mean fracture load shown in Table 4.3 is 1620 N. It is assumed that the uncertainty of applied load is a rectangular distribution. The standard uncertainty in measurement of applied load is then

$$u(W) = (W \cdot 1/100)/\sqrt{3}$$

$$= 9.4 \text{ N}$$

The thickness of the disc plate will be measured with a micrometer. The test method proposed in Section 5.3.1 specifies that the micrometer used shall be accurate to 0.01 mm. In the test performed in this study, the test pieces shall be 2.20 mm in thickness. It is assumed that the distribution of the uncertainty of length measurement is a rectangular distribution. The standard uncertainty in measurement of thickness of the disc plate is then

$$u(t) = 0.01/\sqrt{3}$$

$$= 0.006 \text{ mm}$$

The radius of the support ring will be measured with a micrometer. The test method proposed in Section 5.3.1 specifies that the micrometer used shall be accurate to 0.01 mm. The test method also specifies that the test jig was designed to have a 40 mm ring support diameter. It is assumed that the distribution of the uncertainty of length measurement is a rectangular distribution. The standard uncertainty in measurement of radius of the support ring is then

$$u(R_S) = 0.01 / \sqrt{3}$$

$$= 0.006 \text{ mm}$$

The radius of the disc plate will be measured with a micrometer. The test method proposed in Section 5.3.1 specifies that the micrometer used shall be accurate to 0.01 mm. The test method also specifies that the test pieces shall be 43.00±2.15mm in diameter. It is assumed that the distribution of the uncertainty of length measurement is a rectangular distribution. The standard uncertainty in measurement of radius of the disc plate is then

$$u(R_c) = 0.01 / \sqrt{3}$$

$$= 0.006 \text{ mm}$$

The radius of the load ring will be measured with a micrometer. The test method proposed in Section 5.3.1 specifies that the micrometer used shall be accurate to 0.01 mm. The test method also specifies that the test jig was designed to have a 10 mm load ring diameter. It is assumed that the distribution of the uncertainty of length measurement is a rectangular distribution. The standard uncertainty in measurement of radius of the support ring is then

$$u(R_L) = 0.01 / \sqrt{3}$$

$$= 0.006 \text{ mm}$$

The loading rate, temperature variation, operation conformance and many other random effects will also cause uncertainty during the test. In the test performed in this study, the standard deviation for a set of ring-on-ring tests of

sample size 10 shown in the Table 4.2 is 29 MPa. It is assumed that the uncertainty contributed from random effects is a t-distribution; the standard uncertainty contributed from random effects is then

$$u(\Delta) = s/\sqrt{n}$$

$$= 9.2 \text{ MPa}$$

The combined standard uncertainty, u_c, will be determined from equation (6.9) as follows and shown in Table 6.1.

$$u_c = [(9.4)^2\cdot(0.202)^2 + (0.006)^2\cdot(-297.25)^2 + (0.006)^2\cdot(7.592)^2 + (0.006)^2\cdot(2.23)^2 + (0.006)^2\cdot(-19.5)^2 + (9.2)^2\cdot(1)^2]^{1/2}$$

$$= 9.6 \text{ MPa}$$

Uncertainty source	Uncertainty value	Probability distribution	Coverage factor	Standard uncertainty	Sensitivity coefficient	Combined standard uncertainty
W	1620/100	rectangular	$\sqrt{3}$	9.4	0.202	9.6
t	0.01	rectangular	$\sqrt{3}$	0.006	-297.25	
R_S	0.01	rectangular	$\sqrt{3}$	0.006	7.592	
R_O	0.01	rectangular	$\sqrt{3}$	0.006	2.23	
R_L	0.01	rectangular	$\sqrt{3}$	0.006	-19.5	
Δ	$29/\sqrt{10}$	t-distribution	1	9.2	1	

Table 6.1 The combined standard uncertainty for the ring-on-ring test

The combined standard uncertainty could be multiplied by a coverage factor of k=2, which provides a level of confidence of approximately 95%. The expanded uncertainty (k=2) is therefore 19.2 MPa.

In the test performed in this study, the mean strength for a set of ring-on-ring tests of sample size 10 shown in Table 4.2 is 325 MPa. The uncertainty of

the ring-on-ring test proposed in Section 5.3.1 could be reported as 5.9 % at a confidence level of approximately 95%.

6.4.2 The ball-on-ring test method

The ball-on-ring test involves supporting a disc plate on a ring and centrally loading with a ball. The tensile fracture stress determined by ball-on-ring test is obtained from the fracture load using equation (4.2) shown in Section 4.3.2. For a set of tests by the method proposed in Section 5.32, the equation (4.2) can be rewritten in the same manner as described in Section 6.4.1 as follows

$$\sigma_f = \frac{3W(1+\nu)}{4\pi t^2}\{1 + 2\ln\frac{R_S}{R'_L} + (\frac{1-\nu}{1+\nu})[1 - \frac{R'^2_L}{2R_S{}^2}]\frac{R_S{}^2}{R_O{}^2}\} + \Delta \qquad (6.10)$$

From the equation (6.10), it is known that the flexural strength is a function of the applied load, thickness of the disc plate, Poisson's ratio of the disc plate, contact radius of the ball, radius of the support ring, and radius of the disc plate, and random effect. From the equation (4.3), it is also known that the contact radius of the ball, R'_L, is the function of the applied load, the diameter of the load ball, the Poisson's ratio and Young's modulus of the ball and disc plate. Thus the equation (6.10) can be expressed as

$$\sigma_f = f(W,\ t,\ R_S,\ R_O,\ d,\ \Delta) \qquad (6.11)$$

The major components contributing to the overall uncertainty are:

- $u(W)$: the uncertainty in measurement of the applied load,
- $u(t)$: the uncertainty in measurement of thickness of the disc plate,
- $u(R_s)$: the uncertainty in measurement of radius of the support ring,
- $u(R_o)$: the uncertainty in measurement of radius of the disc plate,
- $u(d)$: the uncertainty in measurement of diameter of the load ball,
- $u(\Delta)$: the uncertainty contributed from random effect.

The uncertainty of the mean biaxial flexural strength then can be expressed as under:

$$u_c^2(\sigma_f) = \left[\frac{\partial\sigma}{\partial W}\right]^2 u^2(W) + \left[\frac{\partial\sigma}{\partial t}\right]^2 u^2(t) + \left[\frac{\partial\sigma}{\partial R_S}\right]^2 u^2(R_S) + \left[\frac{\partial\sigma}{\partial R_O}\right]^2 u^2(R_O)$$
$$+ \left[\frac{\partial\sigma}{\partial d}\right]^2 u^2(d) + \left[\frac{\partial\sigma}{\partial\Delta}\right]^2 u^2(\Delta) \qquad (6.12)$$

The standard uncertainty of each major component and the combined standard uncertainty will be determined in the same manner as described in Section 6.4.1 and shown in Table 6.2.

Uncertainty source	Uncertainty value	Probability distribution	Coverage factor	Standard uncertainty	Sensitivity coefficient	Combined standard uncertainty
W	958/100	rectangular	$\sqrt{3}$	5.5	0.415	16.4
t	0.01	rectangular	$\sqrt{3}$	0.006	-343.3	
R_S	0.01	rectangular	$\sqrt{3}$	0.006	5.12	
R_O	0.01	rectangular	$\sqrt{3}$	0.006	-0.768	
d	0.01	rectangular	$\sqrt{3}$	0.006	-3.97	
Δ	$51/\sqrt{10}$	t-distribution	1	16.1	1	

Table 6.2 The combined standard uncertainty for the ball-on-ring test

The combined standard uncertainty could be multiplied by a coverage factor of k=2, which provides a level of confidence of approximately 95%. The expanded uncertainty (k=2) is therefore 32.8 MPa.

In the test performed in this study, the mean strength for a set of ball-on-ring tests of sample size 10 shown in Table 4.6 is 547 MPa. The uncertainty of a ball-on-ring test proposed in Section 5.3.2 could be reported as 6.0 % at a confidence level of approximately 95%.

6.4.3 The 4-Ball test method

The 4-Ball test involves a ball-loaded disc supported by three equi-spaced balls. The tensile fracture stress determined by 4-Ball test is obtained from the fracture load using equation (4.4) shown in Section 4.4.2. For a set of tests by the method proposed in Section 5.3.3, the equation (4.4) can be rewritten in the same manner as described in Section 6.4.1 as follows

$$\sigma_f = (3\, W_f / 2\pi t^2)\,[(1+\nu)\ln(R_S/R'_L) + (1+\nu)/2 + (1-\nu)\,(2R_S^2 - R'^2_L)/4R_O^2] + \Delta \qquad (6.13)$$

From the equation (6.13), it is known that the flexural strength is a function of the applied load, thickness of the disc plate, Poisson's ratio of the disc plate, contact radius of the ball, radius of pitch circle diameter, radius of the disc plate and random effects. From the equation (4.5), it is also known that the contact radius of the ball, R'_L, is the function of the applied load, the diameter of the ball, the Poisson's ratio and Young's modulus of the ball and disc plate. Thus, the equation (6.13) can be expressed as

$$\sigma_f = f(W,\ t,\ R_s,\ R_o,\ d,\ \Delta) \qquad (6.14)$$

The major components contributing to the overall uncertainty are:

u(W) : the uncertainty in measurement of applied load,
u(t) : the uncertainty in measurement of thickness of the disc plate,
$u(R_s)$: the uncertainty in measurement of radius of pitch circle diameter,
$u(R_o)$: the uncertainty in measurement of radius of the disc plate,
u(d) : the uncertainty in measurement of diameter of the ball,
$u(\Delta)$: the uncertainty contributed from random effects.

The uncertainty of the mean biaxial flexural strength then can be expressed as under:

$$u_c^2(\sigma_f) = \left[\frac{\partial\sigma}{\partial W}\right]^2 u^2(W) + \left[\frac{\partial\sigma}{\partial t}\right]^2 u^2(t) + \left[\frac{\partial\sigma}{\partial R_s}\right]^2 u^2(R_s) + \left[\frac{\partial\sigma}{\partial R_o}\right]^2 u^2(R_o)$$

$$+\left[\frac{\partial\sigma}{\partial d}\right]^2 u^2(d)+\left[\frac{\partial\sigma}{\partial\Delta}\right]^2 u^2(\Delta) \qquad (6.15)$$

The standard uncertainty of each major component and the combined standard uncertainty will be determined in the same manner as described in Section 6.4.1 and shown in Table 6.3.

Uncertainty source	Uncertainty value	Probability distribution	Coverage factor	Standard uncertainty	Sensitivity coefficient	Combined standard uncertainty
W	965/100	rectangular	$\sqrt{3}$	5.6	0.097	6.1
t	0.01	rectangular	$\sqrt{3}$	0.006	-328.76	
R_S	0.01	rectangular	$\sqrt{3}$	0.006	5.135	
R_O	0.01	rectangular	$\sqrt{3}$	0.006	-0.798	
d	0.01	rectangular	$\sqrt{3}$	0.006	3.99	
Δ	$18/\sqrt{10}$	t-distribution	1	5.7	1	

Table 6.3 The combined standard uncertainty for the 4-Ball test

The combined standard uncertainty could be multiplied by a coverage factor of k=2, which provides a level of confidence of approximately 95%. The expanded uncertainty (k=2) is therefore 12.2 MPa.

In the test performed in this study, the mean strength for a set of 4-ball tests of sample size 10 shown in Table 4.10 is 562 MPa. The uncertainty of 4-Ball test proposed in Section 5.3.3 could be reported as 2.2% at a confidence level of approximately 95%.

6.5 Discussion

When reporting the results of a measurement, it is necessary to give some

quantitative indication of the quality of the result be given so that those who are going to use the results can assess its reliability. Without such an indication of the quality, measurement results cannot be compared either among themselves or with reference values given in different kinds of specifications or standards. It is therefore necessary that there is a readily implemented, easily understood, and generally accepted procedure for characterizing the quality of a result of a measurement, that is, for evaluating and expressing its uncertainty.

In many cases, the results of measurements are determined on the basis of repeated observations. There are always variations in repeated observations. How widespread these variations depend on how different or similar the conditions for the repeated observations are. A measurement is said to be precise when there is closeness of agreement between repeated results when the measurements are done under prescribed conditions. Often a large number of significant digits is obtained and there is confidence in the reliability of these digits. Accuracy is a general term which is used in the sense that an accurate measurement is one where the result is believed to be close to the true value. The uncertainty of a measurement is a quantitative measure of how close the measured value is to the true value. It is determined by calculations and estimates after developing a comprehensive model of the measurement and measuring system, taking into account the definition of the measurand and the effects of influential quantities.

Uncertainty means doubt. In its broadest sense, uncertainty of measurement means doubt about the exactness of the results of a measurement and is expressed in terms of the range in which the result may be in error. In general, the result of a measurement is only an approximation or estimate of the value of the measurand and thus is complete only when accompanied by a statement of the uncertainty of that estimate.

The estimation of uncertainty in measurement for the biaxial flexure test standard proposed in this thesis is based on the methodology provided in the ISO Guide to the expression of uncertainty in measurement, which offers a universal method that is internationally recognised and suitable for evaluation and expressing the uncertainty of the result of a measurement.

It was found that the uncertainty of measurement at a confidence level of approximately 95% is about 5.9% in the ring-on-ring test method, 6.0% in the ball-on-ring test method, and 2.2% in the 4-Ball test method. It can be seen that the uncertainty in measurement for the biaxal flexure test standard proposed in this thesis is very low compared to the inherent variability of strength of ceramic materials.

The possible sources of uncertainty in biaxial flexure testing proposed in this thesis include the applied load, thickness of the disc plate, radius of the support ring, radius of the load ring, radius of the disc plate, diameter of the load ball, radius of pitch circle diameter, loading rate, temperature variations, operation conformance and many other random effects.

From the results of estimation, it was found that the applied load, thickness of the disc plate, and random effects are the three major components contributing to the overall uncertainty. The total uncertainty of measurement in biaxial flexure testing can therefore be significantly minimised by the reduction of the uncertainty contributed from these components, especially from random effects.

CHAPTER 7

Conclusion

7.1 Discussion and conclusions

Engineering ceramics are possible to have excellent thermal, mechanical and chemical properties, according to control materials, chemical compositions and processes. With their superior functional traits and special characteristics, engineering ceramics are not only contributing to the invigoration and increasing sophistication of the existing industry, but are also being used as a new material that induces technical innovation in industries at the forefront of these times, such as aviation, space and biotechnology. Recently, engineering ceramics are being looked upon, hopefully, as the material that creates the saving of energy and conservation of resources needed in response to global environmental problems. It is also expected that engineering ceramics broadens the scope of its application, and will gradually spread into our daily lives.

The market growth of engineering ceramics seems to be much more sluggish than was expected over the past ten years. The obstacles to commercialisation of engineering ceramics have been demonstrated. It is found that three major problems existed: Economics, Reliability and Applicability, all would be tackled for more efficient development of the market. It is known that there are many instances where even the cost performance is higher when compared to materials now in use. The lack of reliability in such material properties related to failure or fracturing during service has been found. There also exists an uncertainty about ceramics' applications. To solve the aforementioned problems and to strengthen the promotion of commercialisation, quality standards and standarisation of evaluation methods are proposed as the major issues.

The need for standardisation of engineering ceramics has been discussed in Chapter 1. There are many reasons to establish standards for engineering ceramics. One is the creation of a common language. This will enable a

manufacturer to communicate clearly with a customer's product engineers, designers and purchasing agents anywhere in the world. Another need is to address concerns of public health and safety, where appropriate. This includes the impact on the environment. Perhaps, the primary need met by standards lies in the assurance they provide that a product meet requirements for quality and performance.

Creating standards will not be easy. The field of engineering ceramics is continually changing. Product forms and applications vary widely. Even definitions and terms remain to be established. The concept of standardisation for engineering ceramics is expanding from the simple arrangement of past, completed research activities to the early stage of standardisation of engineering ceramics with new research and development. Active research and development is required to promote early stage standardisation for engineering ceramics.

The present situation of standardisation for engineering ceramics in Japan, the United States, Europe and on an international level has been presented. The Collyear Report in the United Kingdom stressed, some years ago, the importance of test methods standardisation. The development of standard test methods has become one of three main lines of standardisation activities in the United Kingdom.

Strength is one of the most important mechanical properties of engineering ceramics. As a consequence of the cost and difficulty of conducting direct tensile testing on engineering ceramics, the strength of engineering ceramics is often measured by the well-known flexure test method.

Several test techniques for flexure testing of engineering ceramics has been developed. There are many similarities among them. Nevertheless, a myriad of test configurations arose with various specimen sizes and shapes, fixture sizes and types. There is little consistency in procedures or results. In order to obtain more consistent and accurate test results, the standardisation of flexure testing of engineering ceramics must be carried out. The research in this thesis attempts to establish a standard flexure test method which can be easily performed and possesses the accurate and consistent flexure testing results.

The methodology which has been used here and which has been highlighted where appropriate is valid in principle to the generation of other similar standards, e.g., determination of fracture toughness.

Some suitable and commonly used flexure test techniques has been described (See Chapter 2 to 4). These techniques can be grouped into two methods, uniaxial flexure tests and biaxial flexure test.

Uniaxial flexure tests, such as three- or four-point beam bending tests, have long been used to measure ceramic strengths. The specimen in beam bending tests can have a circular, square, or rectangular cross section and is uniform along the complete length. The stress solution for the beam bending tests is known and well developed in the materials' textbooks.

Several types of errors occur in the beam bending tests, which cause the true stress in the outer fibre of the beam to be different from the stress calculated from simple beam theory formulae. These errors are either due to simple beam theory assumptions, or to sources arising from external influences. The limitations of simple beam theory, the most common sources of error arising from external load applications, and the methods for minimization of these errors have been discussed.

Many standards for beam bending tests of engineering ceramics have been established. The standardisation processes differed considerably in different countries, and therefore certain aspects of the standard varied significantly. The comparison of those standards has been outlined.

Biaxial flexure tests have obtained much attention recently since service applications of engineering ceramics generally involve multiaxial loads. These tests also possess the following attractive features:

(1) The disc specimens for the tests are easy to produce.

(2) The tests are simple to perform.

(3) The tests are not critically affected by poor specimen tolerance.

(4) Specimen failure would not be dependent upon edge condition.

There are many possible ways for the biaxial flexure tests. Six such test

methods are the ring-on-ring test, ball-on-ring test, 4-Ball test, piston-on-ring test, piston-on-3ball test, and hydraulic pressure test.

Measurement of flexural strength must be accurate if they are to be really useful and reliable. The variability in flexural strength results is often a consequence of the inherent scatter in tensile strength of brittle ceramics, but it is compounded by experimental errors in strength test methods and often by inconsistency in the materials themselves.

Primary factors which have contributed to the measurement variations of biaxial flexural strengths could be the specimen size, surface finish, fixture geometry, loading rate, and sample size.

The theoretical analysis and experimental investigation of three major techniques of the biaxial flexure tests for engineering ceramics, i.e. ring-on-ring, ball-on-ring, and 4-Ball test have been carried out. The effects of varying the test parameters on flexural strength have been discussed in Chapter 4.

The ring-on-ring test involves supporting a circular plate on a ring and loading with a small concentric ring. The ring-on-ring loading fixture was constructed to provide a biaxial tension strength test in which the effective stressed area or volume of the specimen is comparable to the conventional uniaxial flexure tests, such as four-point beam bend tests. The test is gaining considerable popularity, as an exact analytical stress solution is available for it.

The ball-on-ring test involves supporting a disc plate on a ring and centrally loading with a ball. Its advantages are precise knowledge of the stresses produced in the specimen, simple test fixtures and specimen geometry, and minimum requirements for alignment.

The 4-Ball test involves a ball-loaded disc supported by three equi-spaced balls. The three-ball support is advantageous because it provides kinematic mounting for flat disc specimen. Kinematic mounting cannot easily be achieved when more than three balls are used, which could lead to spurious results. The 4-Ball test is more attractive than the ring-on-ring test, in cases where the disc specimen surfaces are warped or slightly irregular.

The following is a summary of the main findings obtained from the experimental investigation of the effects of the testing parameters on the determination of the fracture strength:

(1) The fixture size of the test jig did strongly influence the fracture strength in the ring-on-ring test. For the same loading rate, the larger the outer ring diameter to the inner ring diameter ratio, the greater the mean fracture strength obtained. The test with the outer ring diameter of 40 mm and inner ring diameter of 10mm showed the smallest value of coefficient of variation.

The dependence of fracture strength on the fixture size was associated with variations in the stress distribution in the test specimen and the flaw size distribution of the material. The area and volume under peak tensile stress or near peak tensile stress is greater for the ring-on-ring test jig with smaller the outer ring diameter to inner ring diameter ratio, and thus the probability of a larger flaw being exposed to high stress is increased. As a result, the fracture strength measured in the ring-on-ring test jig with larger the outer ring diameter to inner ring diameter ratio is greater than that measured in smaller ring diameter ratio. The smallest value of coefficient of variation obtained from the test with the outer ring diameter of 40mm and inner ring diameter of 10mm was attributed to the narrowest flaw size distribution under ring-on-ring test.

(2) The load ball diameter did slightly influence the fracture strength in the ball-on-ring test. The mean fracture strength obtained from the ball-on-ring test using a 5mm load ball diameter was slightly greater than that obtained using a 10mm ball diameter. The coefficient of variation of testing results obtained from a 5mm ball diameter was smaller than that obtained from a 10mm ball diameter.

The observed strength value is dependent on the flaw size distribution of the material and the stress distribution in the test specimen. The surface area and volume of material under 5mm load ball is smaller than that under 10mm ball, therefore, there are smaller area and volume of material under peak tensile stress and narrower size distribution of the flaws of material in the ball-on-ring test using a 5mm load ball. This leads to the greater fracture strength and the

smaller coefficient of variation of tests obtained from a 5 mm load ball.

(3) The loading rate did strongly influence the fracture strength in the 4-Ball test. For the same pitch circle diameter and same ball diameter, the slower the speed of loading, the smaller the mean fracture strength obtained. However, the loading rate of 0.5 mm/min was found to have the smallest value of coefficient of variation.

The rate of loading can have an effect on fracture strength as the result of stress corrosion mechanisms, particularly at low strain rates. In general, the slower the rate of loading, the greater the opportunity for stress corrosion phenomena to weaken the specimen. Selection of the loading rate may have to be determined by experiment, depending on the elastic compliance of the test machine, the stiffness of the test jig and the elastic properties of the test specimen. The experimental results shows that the loading rate of 0.5 mm/min has the smallest value of coefficient of variation for the test equipment and test material presented in this thesis.

(4) The pitch circle diameter of the test jig did not strongly influence the fracture strength. The mean fracture strength obtained from 4-Ball test using 30mm pitch circle diameter did not differ significantly from that obtained using 40 mm pitch circle diameter. However, the coefficient of variation of testing results obtained from a 30mm pitch circle diameter was smaller than that obtained from a 40mm pitch circle diameter.

With the same load ball diameter and same loading rate, the area and volume of material under peak tensile stress was similar for the 4-Ball test using 30 mm pitch circle diameter and 40mm pitch circle diameter, and thus the measured fracture strength did not differ significantly. The narrower distribution of flaw size in the specimen volume under 30 mm pitch circle diameter is the main reason to cause its smaller value of the coefficient of variation.

(5) The ball diameter did slightly influence the fracture strength in the 4-Ball test. The mean fracture strength obtained using a 5mm ball diameter was slightly greater than that obtained using a 10mm ball diameter. The mean fracture strength values from the two sets of test differed by only 2.7%.

The coefficient of the variation of testing results obtained from a 5 mm ball diameter was smaller than that obtained from a 10 mm ball diameter.

The same reason as described in conclusion (2) can be applied to explain this conclusion.

(6) For the same support ring diameter and same loading rate, the mean fracture strength obtained from the two test methods (ring-on-ring and ball-on-ring) is dependent of the nature of the load. The disc specimens loaded by a ball was found to have larger fracture strength and a smaller coefficient of variation than that loaded by a ring.

The area and volume under peak tensile stress or near peak tensile stress is much greater for ring-on-ring test than for 4-Ball test, and thus the probability of a larger flaw being exposed to high stress is increased. As a result, the fracture strength measured in ring-on-ring test is lower than that measured in 4-Ball test. The smaller value of coefficient of variation obtained from the 4-Ball test was attributed to the narrower flaw size distribution of the material.

(7) For the same support diameter and same load ball diameter, the mean fracture strength obtained from the two test methods (ball-on-ring and 4-Ball) is independent of the nature of the support.

With the same support diameter and same load ball diameter, there was no difference for the area and volume of material under peak tensile stress between the two test methods, and thus the measured fracture strength did not differ significantly.

The ultimate objective of the work in this thesis is to develop a methodology whereby the flexure test method standards can be formulated in order to improve the accuracy and consistency of flexure testing results of engineering ceramics. Based on the methodology and the results of the theoretical analysis and experimental investigation of three major techniques of the biaxial flexure tests for engineering ceramics, the important characteristic features governing flexure testing have been determined and a draft standard has been proposed by this thesis in Chapter 5.

The draft standard presented in this thesis with the provisional title of "Engineering ceramics–Determination of biaxial flexural strength at room temperature" consists of three parts:

Part1: The ring-on-ring test method

Part 2: The ball-on-ring test method

Part 3: The 4-Ball test method

This draft standard covers the three major testing methods for determining the biaxial flexural strength (modulus of rupture) of engineering ceramics. The ring-on-ring, ball-on-ring and 4-Ball test fixtures were all adopted as standard, since it is the fact that each of these systems is suited for a particular application and each has different advantages and disadvantages.

Each part of this draft standard contains the following content: scope, normative references, definitions, significance and use, apparatus, test pieces, test procedure, calculations, and test report.

This draft standard, developed for satisfying the need for standardisation of test methods for flexural strength of engineering ceramics, is intended for use by manufacturers and purchasers of engineering ceramics to be used as high strength materials of machine parts, structural materials etc. for material development, quality control, characterisation and design data generation purposes.

The use of this draft standard test method will bring some consistency to the flexural strength testing of engineering ceramics. Comparative analysis of data will be more meaningful. Significant experimental errors will be minimised. It is expected that the use of this draft standard will permit the generation of high quality and reproducible design data.

The flexural strength of engineering ceramics is not a deterministic quantity, but will vary from one specimen to another. There will be an inherent statistical scatter in the results for finite sample sizes. The three major biaxial test methods prescribed in this draft standard has been devised so that the precision is very high and the bias very low compared to the inherent variability

of strength of the material. However, the uncertainty of measurement in flexure testing always exists and needs to be estimated.

The uncertainty of a measurement is a parameter, associated with the result of a measurement, that characterizes the dispersion of the values that could reasonably be attributed to the measurand. After all known corrections have been applied, there remains some dispersion which is associated with the measured value. The uncertainty statement quantifies this dispersion. The uncertainty of a measurement is determined by calculations and estimates after developing a comprehensive model of the measurement and measuring system, taking into account the definition of the measurand and the effects of influence quantities.

Since the clause 5.4.6.2 of ISO/IEC 17025 specifies that testing laboratories shall have and apply procedures for estimating uncertainty of measurement, and indicates that in those cases where a well-recognized test method specifies limits to the values of the major sources of uncertainty of measurement and specifies the form of presentation of calculated results, the laboratory is considered to have satisfied this clause by following the test method and reporting instructions, and since the uncertainty of measurement in biaxial flexure testing has never been assessed before, this thesis is a first attempt to identify the components of uncertainty and make a reasonable estimation of uncertainty of measurement in flexure testing.

The estimation of uncertainty of measurement in flexure testing in this work is based on the approach to expressing uncertainty provided in the "Guide to the Expression of Uncertainty in Measurement" published by the ISO. The procedure and results of the estimations of uncertainty in measurement for the biaxial flexure test standard proposed in this thesis have been discussed in Chapter 6.

It is found that the uncertainty of measurement at a confidence level of approximately 95% is about 5.9% in the ring-on-ring test method, 6.0% in the ball-on-ring test method, and 2.2% in the 4-Ball test method. It can be seen that the uncertainty in measurement for the biaxial flexure test standard proposed in this thesis is very low compared to the inherent variability of

strength of ceramic materials.

It is also found that the applied load, thickness of the disc plate, and random effects are the three major components contributing to the overall uncertainty. The total uncertainty of measurement in biaxial flexure testing can therefore be significantly minimised by the reduction of the uncertainty contributed from these components, especially from random effects.

In general, it is very difficult to develop the best appropriate standard for the biaxial flexure testing of engineering ceramics because of the lack of knowledge in such test method. Very few researcher have dealt with the development of this testing technique and the related standardisation activities. This thesis have therefore attempted to give some contributions on developing the methodology for the formulation of standards, identifying the important characteristic features governing flexure testing, establishing the appropriate standards for the flexure test method which can be easily performed and possesses accurate and consistent testing results, and estimating the uncertainty of measurement for the standard flexure test method.

In summary, two basic subjects have been addressed in this thesis, the need for ceramics with their associated problems and standardisation for the 21st century. These topics have been brought together whereby the methodology for the formulation of the document standard is developed and a draft standard is formulated to meet new requirements and to avoid old problems associated with engineering ceramics. Many significant flexure testing techniques have been analysed and their critical parameters identified. This thesis consolidates and integrates the concept of standardisation of flexure testing of engineering ceramics and hopefully will be useful elsewhere. A draft standard for determination of biaxial flexural strength of engineering ceramics at room temperature has been proposed with the estimation of uncertainty of measurement. This draft standard is the underlying theme of this thesis. This draft standard covers the three major testing methods for determining the biaxial flexural strength. These testing methods have been devised so that more consistent and accurate test results can be obtained. The uncertainty in measurement for these standard flexure test methods is very low compared to the inherent variability of the strength of ceramic materials. It is hoped that the

work presented here is a useful contribution, that it pushes forward the frontier of knowledge in flexure testing of engineering ceramics, and that the draft standard can be offered to the British Standards Institution, the European Committee for Standardisation and the International Organization for Standardisation for the establishment of BS, EN and ISO standards.

7.2 Suggestions for further work

The suggestions for further work are twofold: (1) intra-laboratory comparison of strength values, and (2) development of the hydraulic pressure test method standard.

In many cases, the test results are determined on the basis of repeated observations. There are always variations in repeated observations. How widespread these variations are depend on how different or similar the conditions for the repeated observations are.

A series of observations can be made by the same test methods, by a single analyst carrying out several determinations, at the same time, with the same equipment, under constant conditions in the same laboratory. It is expected that the smallest variations in repeated observations will be obtained under such conditions. However, a series of observations can also be carried out under changed conditions such as different test methods, different analysts, different measuring instruments and test equipment at "reproducibility conditions". The variations in repeated observations will naturally increase in such circumstances.

If the same kind of test is performed in different laboratories in different countries there is a risk that the conditions for the tests might be increasingly different. If there is an interest to compare test results between countries or to accept test results from laboratories in other countries, it is important that certain safeguards are in place to minimise reproducibility factors.

For the purpose of setting up a standard flexure test method which possesses the reproducibility of testing results, it is desired to carry out the intra-laboratory comparison of strength values measured in different laboratories by the same test method proposed in this thesis.

The second further work would involve the development of the hydraulic pressure test method standard. The test consists of loading a disc specimen, which is supported along a concentric line support near its periphery, with lateral uniform pressure. It was apparently first used in the British glass industry for strength testing of plate glass. More recently, it has been used to evaluate impact damage and erosion parameters of brittle materials.

The hydraulic pressure loading test has proved to be of great practical value. The main advantages of its use can be summarized [91–93]:

(1) Edge failures which cause difficulty with tensile testing or three- or four-point bending are largely eliminated.

(2) The test region extends almost to the edge of the specimen, in contrast to the ring-on-ring test where only a small area within the inner ring is suitable.

(3) There is little stress concentration from mechanical pressure.

(4) There is much less effect due to warped plates.

(5) Small specimens can be used, and the method can easily be further miniaturized.

(6) The method is extremely rapid in operation.

(7) It possesses a circularly symmetrical stress field.

The hydraulic pressure loading test has shown advantages over the standard biaxial flexure test method in some situations. However, the method has not yet received widespread acceptance as a fundamental material test. It needs to be further investigated and standardised by the development of test method standards, in order to, improve the accuracy and consistency of flexure testing results of engineering ceramics.

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Appendix 1

The programmes of working groups in CEN/TC 184

For WG 1 (Classification), the only work item is a classification scheme by which advanced technical ceramics may be classified by the following:

1. Application: Electrical, electronic, mechanical, thermal, biomedical, magnetic, nuclear , optical and thermomechanical.
2. Chemical nature: Precursor, powder, solid ceramic.
3. Processing method: Preparation of precursors and powders, shaping, processes, finishing processes and others.
4. Property data: Property type, property and range.

The Scheme is machine readable and therefore suitable for any database system.

WG 2 (Powders) concerned with the following work programme:

1. Determination of impurities in alumina—AAS and ICP, Pr En 725-1.
2. Determination of impurities in barium titanate powders—Pr EN 725-2.
3. Determination of oxygen content in non-oxide ceramics—Pr EN 725-3.
4. Determination of grain size distribution—Particle size, Pr EN 725-5.
5. Determination of specific surface area—BET method, Pr EN 725-6.
6. Determination of bulk density—Tapped method Pr EN725-8, Untamped method Pr EN 725-9.
7. Determination of absolute density—Pycknometric method, Pr EN 725-7.
8. Determination of compaction properties—Pressing trials, Pr EN 725-10.
9. Determination of sintering curve—pr EN 725

WG 3 (Monolithic Ceramics) concerned with the following work programme:

1. Sampling and testing
2. Determination of the presence of cracks and other defects by dye penetration tests—(EN) Pr EN 623-1.
3. Determination of density and porosity—Archimedian and dimensional methods (EN).

4. Determination of grain size—Based on ASTM E112 for metals (ENV).
5. Determination of surface finish—Based on contacting surface profilometry (ENV).
6. Determination of short term flexural strength at RT—Based on MIL standard jig and sample specific, 3 and 4 point bending, 3 surface finishes, (EN) Pr EN 843-1.
7. Determination of elastic properties at RT—4 methods: bending, resonance, impulse excitation and ultrasonic (ENV).
8. Determination of sub-critical crack growth by constant stressing rate tests.
9. Measurement of hardness—Rockwell, Vickers HV 1.0 and Knoop (ENV).
10. Determination of short-term strength at elevated temperatures (ENV).
11. Determination of pyroplastic deformation—Self-loaded deformation (ENV).
12. Guidelines to thermal shock test—Based on crack detection and strength reduction (ENV).
13. Determination of thermal expansion—2 grades A/B defines apparatus and calibration etc. (EN) Pr EN 821-1.
14. Determination of thermal diffusivity—Flash method laser and heat pulse, (EN) Pr EN 821-2.
15. Determination of specific heat—Based on drop calorimetry and DSC (ENV).

WG4 (Ceramic Composites) concerned with the following work programme:

1. Tensile strength at RT—Standard tensile test describes fibre mounting requires fibre diameter evaluation.
2. Compressive strength at RT.
3. Flexural strength at RT.
4. Shear strength at RT—Based on double punch shearing a specimen through a die, 3 point bending strength , compression test on notched specimens.
5. Determination of thermal expansion.
6. Determination of thermal diffusivity.
7. Determination of thermal conductivity—Based on flash diffusivity /specific heat

8. Determination of specific heat.
9. Determination of density—Archimedian and dimensional methods.
10. Determination of size content—Measures %wt loss by solvent extraction.
11. Determination of linear mass—Measures the mass per unit length.
12. Determination of filament diameter—Optical and laser interference methods.
13. Determination of tensile strength of filament at RT.

WG5 (Ceramic Coatings) concerned with the following work programme:

1. Definitions of Thin/Thick coatings.
2. Sampling.
3. Determination of chemical composition—Based on EPMA.
4. Determination of coating thickness—Based on contact profilometry and a cap grinding method.
5. Characterization of coating morphology.
6. Characterization of microstructure.
7. Characterization of adhesion—Based on a scratch test.
8. Determination of coating hardness.
9. Determination of elastic constants.
10. Determination of elastic constants.
11. Quasi-static tests of mechanical properties.
12. Fatigue properties.
13. Thermal shock resistance.
14. Determination of thermal stresses/strains.
15. Wear resistance.
16. Corrosion resistance.

Appendix 2

The projects in development in ISO/TC 206

The number and title of projects in development in ISO/TC 206 are listed below [27]:

ISO/DIS 14703: Fine ceramics (Advanced ceramics, Advanced technical ceramics)

—Sample preparation for the determination of particle size distribution of ceramic powders

ISO/DIS 14704: Fine ceramics (Advanced ceramics, Advanced technical ceramics)

—Test method for flexural strength of monolithic ceramics at room temperature

ISO/DIS 14705: Fine ceramics (Advanced ceramics, Advanced technical ceramics)

—Test method for hardness of monolithic ceramics at room temperature

ISO/WD 15165: Fine ceramics (Advanced ceramics, Advanced technical ceramics)

—Classification system

ISO/WD 15490: Fine ceramics (Advanced ceramics, Advanced technical ceramics)

—Test method for tensile strength of monolithic ceramics at room temperature

ISO/WD 15732: Fine ceramics (Advanced ceramics, Advanced technical ceramics)

—Test method for fracture toughness of monolithic ceramics at room temperature by single edge pre-cracked beam (SEPB) method

ISO/WD 15733: Fine ceramics (Advanced ceramics, Advanced technical ceramics)

—Test method for tensile stress-strain behaviour of continuous fibre-reinforced composites at room temperature

NP 5: Fine ceramics (Advanced ceramics, Advanced technical ceramics)
—Determination of specific surface area of ceramic powders by the gas absorption using the BET method

NP 8: Fine ceramics (Advanced ceramics, Advanced technical ceramics)
—Test method for flexural strength of monolithic ceramics at elevated temperatures

NP 10: Fine ceramics (Advanced ceramics, Advanced technical ceramics)
—Test method for elastic moduli of monolithic ceramics at room temperature

NP 11: Fine ceramics (Advanced ceramics, Advanced technical ceramics)
—Weibull statistics of strength data

NP 12: Fine ceramics (Advanced ceramics, Advanced technical ceramics)
—Test method for thermal expansion of monolithic ceramics by dilatometry technique

PWI 2: Fine ceramics (Advanced ceramics, Advanced technical ceramics)
—Determination of particle size distribution of ceramic powders by laser diffraction method.

Appendix 3

Stress distribution of ring-on-ring test

Consider a thin circular disc, simply supported and loaded with a ring as shown in Fig. A3.1.

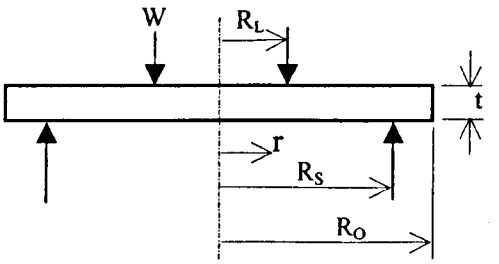
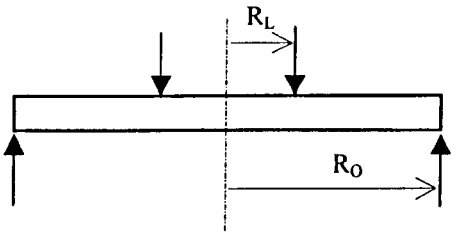


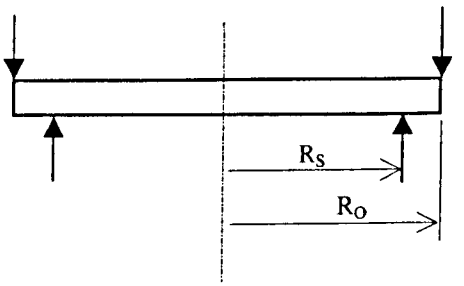
Fig. A3.1

The above loading can be taken as the super position of the following two loadings.

(i)



(ii)



The loading giving by (i) and (ii) has a standard closed form analytical solutions for deflection [81]. A summation of the two values gives the deflection for the disc specimen shown in Fig.A3.1. The deflection, ω, at a general radius r is given as follows.

For $r \le R_L$:

$$\omega = \frac{W}{8\pi D}\{(R_L^2 + r^2)\ln\frac{R_L}{R_o} - (R_S^2 + r^2)\ln\frac{R_S}{R_O} + (R_S^2 - R_L^2)[\frac{(3+\nu)R_O^2 - (1-\nu)r^2}{2(1+\nu)R_O^2}]\}$$

For $R_S \ge r \ge R_L$:

$$\omega = \frac{W}{8\pi D}\{(R_L^2 + r^2)\ln\frac{r}{R_o} - (R_S^2 + r^2)\ln\frac{R_S}{R_O} + (R_O^2 - r^2)[1 + \frac{(1-\nu)}{2(1+\nu)}\frac{R_O^2 - R_L^2}{R_O^2}]$$
$$-(\frac{R_O^2 - R_L^2}{R_O^2})[\frac{(3+\nu)R_O^2 - (1-\nu)r^2}{2(1+\nu)}]\}$$

Where $D = \frac{Et^3}{12(1-\nu^2)}$

The bending moments at a general radius, *r*, can be determined from the deflections by using the standard relations for the tangential, M_t, and, radial, M_r, bending moments, i.e.

$$M_r = -D(\frac{\partial^2 \omega}{\partial r^2} + \frac{\nu}{r}\cdot\frac{\partial \omega}{\partial r})$$

and $$M_t = -D(\frac{1}{r}\frac{\partial \omega}{\partial r} + \nu\cdot\frac{\partial^2 \omega}{\partial r^2})$$

The bending moments for $r \le R_L$ are given by,

$$M_r = \frac{W}{4\pi}[(1+\nu)\ln\frac{R_S}{R_L} + (1-\nu)(R_S^2 - R_L^2)/2R_O^2]$$

$$M_t = M_r$$

(i.e. constant equi-biaxial stressing)

The bending moments for $R_S \ge r \ge R_L$ are given by,

$$M_r = \frac{W}{4\pi}[(1+\nu)\ln\frac{R_S}{r} + (\frac{1-\nu}{2})(\frac{R_L^2}{r^2} - 1) + (1-\nu)(R_S^2 - R_L^2)/2R_O^2]$$

$$M_t = \frac{W}{4\pi}[(1+\nu)\ln\frac{R_S}{R_O} - (\frac{1-\nu}{2})(\frac{R_L^2}{r^2} - 1) + (1-\nu)(R_S^2 - R_L^2)/2R_O^2]$$

The hoop, σ_t, and radial, σ_r, stresses are the principal stresses in the disc. Since the disc is thin they are assumed to vary in magnitude linearly with distance through the disc. At the surface the stresses are given by,

$$\sigma_r = 6M_r / t^2$$

and $$\sigma_t = 6M_t / t^2$$

The maximum bending moment, M_{max}, occurs at $r \leq R_L$, i.e.

$$M_{\max} = \frac{W}{4\pi}[(1+\nu)\ln\frac{R_S}{R_L} + (1-\nu)(R_S^2 - R_L^2)/2R_O^2]$$

The maximum bending stresses, σ_{max}, is then

$$\begin{aligned}\sigma_{\max} &= 6M_{\max}/t^2 \\ &= (3W/2\pi t^2)[(1+\nu)\ln\frac{R_S}{R_L} + (1-\nu)(R_S^2 - R_L^2)/2R_O^2]\end{aligned}$$

Appendix 4

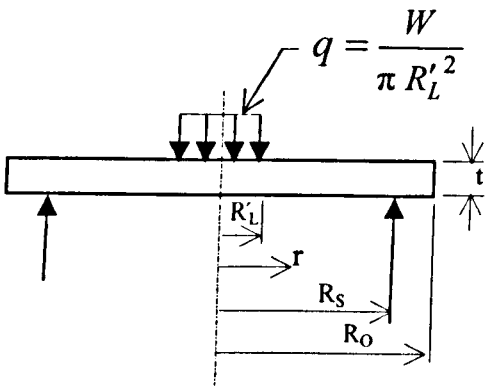
Fracture results of discs in ring-on-ring test

Model No.	Specimen No.	Specimen dia. (mm)	Specimen thickness (mm)	Fracture load (N)	Fracture strength (MPa)
1 outer ring dia. = 40mm inner ring dia. = 10mm	H 1	43.35	2.17	1515	314
	H 2	43.38	2.25	1588	306
	H 3	43.58	2.23	1865	365
	H 4	43.68	2.25	1613	310
	H 5	43.19	2.20	1491	301
	H 6	43.56	2.17	1725	357
	H 7	43.50	2.17	1539	318
	H 8	43.62	2.16	1595	333
	H 9	43.35	2.26	1915	366
	H 10	42.53	2.16	1352	284
2 outer ring dia. = 30mm inner ring dia. = 10mm	I 1	42.36	2.23	1906	284
	I 2	43.46	2.22	2174	325
	I 3	43.48	2.25	2587	376
	I 4	43.30	2.16	1705	269
	I 5	42.28	2.23	1987	296
	I 6	43.36	2.25	2313	336
	I 7	42.35	2.16	1343	213
	I 8	43.41	2.16	1959	309
	I 9	42.48	2.16	1540	244
	I 10	43.40	2.15	1939	309
3 outer ring dia. = 40mm inner ring dia. = 20mm	J 1	43.30	2.15	1783	204
	J 2	43.85	2.15	2062	235
	J 3	43.26	2.16	1986	226
	J 4	43.52	2.14	2055	237
	J 5	42.92	2.16	2324	265
	J 6	43.36	2.15	2197	252
	J 7	43.45	2.15	1871	214
	J 8	43.55	2.15	1580	181
	J 9	43.37	2.15	2038	234
	J 10	43.38	2.16	2166	246

Appendix 5

Stress distribution of ball-on-ring test

Consider a thin circular disc loaded as follows,



Timoshenko [81] gives the differential equation for the deflection, ω, of a thin circular disc subject to a uniformly distributed pressure load:

$$\frac{d}{dr}[\frac{1}{r}\frac{d}{dr}(r\frac{d\omega}{dr})] = \frac{Q}{D} \qquad \text{(A5.1)}$$

Where r is a general radius, Q is the shear force, and D is the flexure rigidity defined by

$$D = \frac{Et^3}{12(1-\nu^2)}$$

Where E is Young's modulus, ν is poisson's ratio, and t is thickness of disc specimen.

For $0 \le r \le R_L'$

$$q\pi r^2 = 2\pi r Q$$

$$Q = \frac{qr}{2} = \frac{Wr}{2\pi R_L'^2}$$

Equation (A5.1) becomes

$$\frac{d}{dr}[\frac{1}{r}\frac{d}{dr}(r\frac{d\omega}{dr})] = \frac{W}{D}\cdot\frac{r}{2\pi R_L'^2}$$

Let ω (r = 0) = 0 , the deflection is given by

$$\omega_1 = \frac{W}{D}\left(\frac{r^4}{64\,\pi R_L'^2} + C_{11}\, r^2\right)$$

for $R_s \geq r \geq R'_L$

$$\frac{d}{dr}\left[\frac{1}{r}\frac{d}{dr}\left(r\frac{d\omega}{dr}\right)\right] = \frac{W}{D2\pi r}$$

The deflection is given by

$$\omega_2 = \frac{W}{D}\left(\frac{1}{8\pi} r^2 \ln r + C_{21}\, r^2 + C_{22} \ln r + C_{23}\right)$$

for $R_o \geq r \geq R_s$

$$\frac{d}{dr}\left[\frac{1}{r}\frac{d}{dr}\left(r\frac{d\omega}{dr}\right)\right] = 0$$

The deflection is given by

$$\omega_3 = \frac{W}{D}\left(C_{31}\, r^2 + C_{32} \ln r + C_{33}\right)$$

The bending moments at a general radius, r, can be determined from the deflections by using the standard relations for the tangential, M_t, and, radial, M_r, bending moments, i.e.

$$M_r = D\,(K_r + \nu\, K_t)$$

$$= -D\left(\frac{d^2\omega}{dr^2} + \frac{\nu}{r}\cdot\frac{d\,\omega}{dr}\right)$$

$$M_t = D\,(K_t + \nu \cdot K_r)$$

$$= -D\left(\frac{1}{r}\frac{d\omega}{dr} + \nu\cdot\frac{d^2\,\omega}{dr^2}\right)$$

The bending moments for $0 \leq r \leq R'_L$ are given by,

$$M_{r1} = -W\left(\frac{r^2}{16\pi\, R_L'^2}(3+\nu) + 2C_{11}\,(1+\nu)\right)$$

and $$M_{t1} = -W\left(\frac{r^2}{16\pi\, R_L'^2}(1+3\nu) + 2C_{11}\,(1+\nu)\right)$$

The bending moments for $R_S \geq r \geq R'_L$ are given by,

$$M_{r2} = -W\left\{\frac{1}{8\pi}\left[2\ln r(1+\nu) + (3+\nu)\right] + 2C_{21}\,(1+\nu) - \frac{C_{22}}{r^2}(1-\nu)\right\}$$

and $$M_{t2} = -W\left\{\frac{1}{8\pi}[2\ln r(1+\nu)+(1+3\nu)]+2C_{21}(1+\nu)+\frac{C_{22}}{r^2}(1-\nu)\right\}$$

The bending moments for $R_O \geq r \geq R_S$ are given by,

$$M_{r3} = -W\left[2C_{31}(1+\nu)-\frac{C_{32}}{r^2}(1-\nu)\right]$$

and $$M_{t3} = -W\left[2C_{31}(1+\nu)+\frac{C_{32}}{r^2}(1-\nu)\right]$$

By using the boundary condition at r = 0

$$\omega_1 = 0 \qquad d\omega_1/dr = 0$$

it is found that, $C_{12} = C_{13} = 0$

By using the boundary condition at $r = R_O$

$$M_{r3}\,(r = R_O) = 0$$

it is found that,

$$(2+2\nu)C_{31} - \left(\frac{1}{R_o^2}-\frac{\nu}{R_o^2}\right)C_{32} = 0$$

For continuity condition at $r = R'_L$

$$\omega_1(r=R'_L) = \omega_2\,(r=R'_L)$$

$$M_{r1} = M_{r2}$$

For continuity condition at $r = R_s$

$$\omega_2\,(r = R_s) = \omega_3\,(r = R_s)$$

$$M_{r2} = M_{r3}$$

From the above requirement for continuity condition, it is found that the bending moments for $r \leq R'_L$ are given by,

$$M_r = \frac{W}{4\pi}\left[(1+\nu)\ln\frac{R_S}{R'_L}+\frac{1+\nu}{2}+\frac{(1-\nu)(2R_S^2-R_L'^2)}{4R_O^2}-\frac{r^2}{4R_L'^2}(3+\nu)\right]$$

and $$M_t = \frac{W}{4\pi}\left[(1+\nu)\ln\frac{R_S}{R'_L}+\frac{1+\nu}{2}+\frac{(1-\nu)(2R_S^2-R_L'^2)}{4R_O^2}-\frac{r^2}{4R_L'^2}(1+3\nu)\right]$$

The maximum bending moment, M_{max}, occurs at r = 0, i.e.

$$M_{\max} = \frac{W}{4\pi}[(1+\nu)\ln\frac{R_S}{R'_L} + \frac{1+\nu}{2} + (1-\nu)(2R_S^2 - R_L'^2)/4R_O^2]$$

The hoop, σ_t, and radial, σ_r, stresses are the principal stresses in the disc. Since the disc is thin they are assumed to vary in magnitude linearly with distance through the disc. At the surface the stresses are given by,

$$\sigma_r = 6M_r / t^2$$

and $$\sigma_t = 6M_t / t^2$$

The maximum bending stresses, σ_{max}, is then

$$\sigma_{\max} = \frac{6M_{\max}}{t^2}$$

$$= \frac{3W(1+\nu)}{4\pi t^2}\left\{1 + 2\ln\frac{R_S}{R'_L} + \left(\frac{1-\nu}{1+\nu}\right)\left(1 - \frac{R_L'^2}{2R_S^2}\right)\frac{R_S^2}{R_O^2}\right\}$$

Appendix 6

Fracture results of discs in ball-on-ring test

Model No.	Specimen No.	Specimen dia. (mm)	Specimen thickness (mm)	Fracture load (N)	Fracture strength (Mpa)
1 ball-on-ring test support ring dia. = 30mm load ball dia. = 10mm loading rate = 0.5mm/min	K 1	43.61	2.21	1051	588
	K 2	43.56	2.16	974	575
	K 3	43.68	2.18	984	569
	K 4	43.37	2.13	974	591
	K 5	43.49	2.23	1024	565
	K 6	43.52	2..27	1002	534
	K 7	42.52	2.18	825	484
	K 8	42.80	2.15	882	530
	K 9	43.77	2.22	1070	593
	K 10	42.31	2.25	798	441
2 ball-on-ring test support ring dia. = 30mm load ball dia. = 5mm loading rate = 0.5mm/min	L 1	43.37	2.17	910	562
	L 2	43.58	2.23	945	551
	L 3	42.36	2.25	935	536
	L 4	43.52	2.15	847	535
	L 5	43.48	2.20	930	557
	L 6	43.25	2.23	956	557
	L 7	43.59	2.16	941	584
	L 8	43.32	2.23	1002	582
	L 9	42.37	2.26	895	510
	L 10	43.47	2.23	1021	592
3 ring-on-ring test support ring dia. = 30mm load ring dia. = 10mm loading rate = 0.5mm/min	I 1	42.36	2.23	1906	284
	I 2	43.46	2.22	2174	325
	I 3	43.48	2.25	2587	376
	I 4	43.30	2.16	1705	269
	I 5	42.28	2.23	1987	296
	I 6	43.36	2.25	2313	336
	I 7	42.35	2.16	1343	213
	I 8	43.41	2.16	1959	309
	I 9	42.48	2.16	1540	244
	I 10	43.40	2.15	1939	309
4 4-Ball test pitch circle dia. = 30mm load ball dia. = 5mm loading rate = 0.5mm/min	F 1	43.44	2.15	924	580
	F 2	42.32	2.22	874	517
	F 3	42.34	2.15	830	526
	F 4	42.22	2.12	715	471
	F 5	43.01	2..10	902	595
	F 6	43.41	2.14	941	596
	F 7	43.34	2.13	911	589
	F 8	42.31	2.10	783	522
	F 9	43.56	2.21	971	575
	F 10	43.36	2.13	911	583

Appendix 7

Stress distribution of 4-Ball test

Consider a thin circular disc, simply supported and loaded with a uniformly distributed pressure as shown in Fig. A7.1.

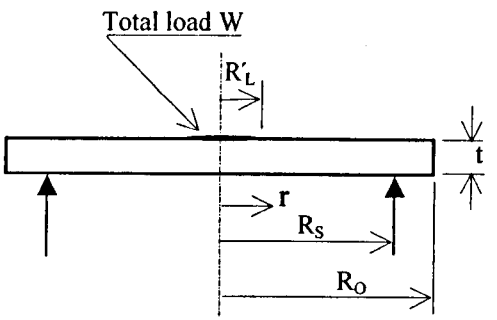
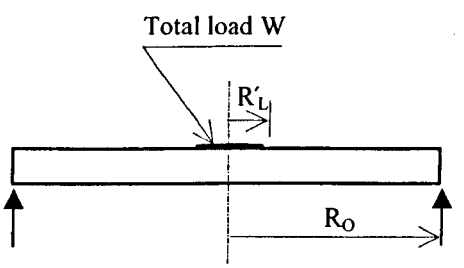


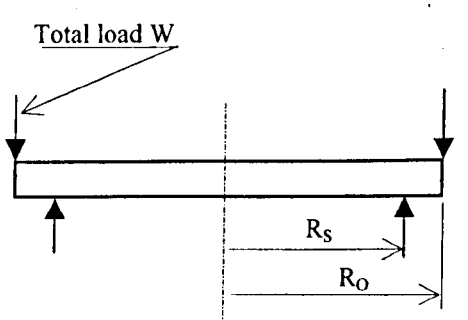
Fig. A7.1

The above loading can be taken as the super position of the following two loadings [12].

(i)



(ii)



Both (i) and (ii) have standard analytical solutions for deflection [81]. A summation of the two values gives the deflection for the disc specimen shown in Fig.A7.1. The deflection, ω, at a general radius r is given as follows:

For $r \le R'_L$

$$\omega = \frac{W}{16\pi D}\{R_L'^2 \ln\frac{R_L'}{R_o} - \frac{R_L'^2}{4} + 2r^2 \ln\frac{R_L'}{R_O} - r^2 + (2R_S^2 - R_L'^2)[\frac{(3+\nu)R_O^2 - (1-\nu)r^2}{2(1+\nu)R_O^2}]$$
$$+ (\frac{r^2}{2R_L'})^2 - 2(R_S^2 + r^2)\ln\frac{R_S}{R_O}\}$$

Where $D = \frac{Et^3}{12(1-\nu^2)}$

For $R_S \ge r \ge R'_L$

$$\omega = \frac{W}{16\pi D}\{R_L'^2 \ln\frac{r}{R_o} + 2r^2 \ln\frac{r}{R_O} + (R_S^2 - r^2)[\frac{(3+\upsilon)}{(1+\nu)}] - 2(R_S^2 + r^2)\ln\frac{R_S}{R_O}$$
$$+ (\frac{(1+\nu)}{2R_O{}^2(1+\nu)})(-2r^2R_S^2 - R_L'^2R_O{}^2 + r^2R_L'^2 + 2r^2R_O{}^2)\}$$

The bending moments at a general radius, r, can be determined from the deflections by using the standard relations for the tangential, M_t, and, radial, M_r, bending moments, i.e.

$$M_r = -D(\frac{\partial^2\omega}{\partial r^2} + \frac{\nu}{r}\cdot\frac{\partial\omega}{\partial r})$$

and $$M_t = -D(\frac{1}{r}\frac{\partial\omega}{\partial r} + \nu\cdot\frac{\partial^2\omega}{\partial r^2})$$

The bending moments for $r \le R'_L$ are given by,

$$M_r = \frac{W}{4\pi}[(1+\nu)\ln\frac{R_S}{R_L'} + \frac{1+\nu}{2} + \frac{(1-\nu)(2R_S^2 - R_L'^2)}{4R_O^2} - \frac{r^2}{4R_L'^2}(3+\nu)]$$

$$M_t = \frac{W}{4\pi}[(1+\nu)\ln\frac{R_S}{R_L'} + \frac{1+\nu}{2} + \frac{(1-\nu)(2R_S^2 - R_L'^2)}{4R_O^2} - \frac{r^2}{4R_L'^2}(1+3\nu)]$$

The bending moments for $R_S \ge r \ge R'_L$ are given by,

$$M_r = \frac{W}{4\pi}[(1+\nu)\ln\frac{R_S}{r} - \frac{1-\nu}{2} + \frac{(1-\nu)(2R_S^2 - R_L'^2)}{4R_O^2} + \frac{(1-\nu)R_L'^2}{4r^2}]$$

and $$M_t = \frac{W}{4\pi}[(1+\nu)\ln\frac{R_S}{r} + \frac{1-\nu}{2} + \frac{(1-\nu)(2R_S^2 - R_L'^2)}{4R_O^2} - \frac{(1-\nu)R_L'^2}{4r^2}]$$

The hoop, σ_t, and radial, σ_r, stresses are the principal stresses in the disc. Since the disc is thin they are assumed to vary in magnitude linearly with distance through the disc. At the surface the stresses are given by,

$$\sigma_r = 6M_r / t^2$$

and $$\sigma_t = 6M_t / t^2$$

The maximum bending moment, M_{max}, occurs at r = 0 , i.e.

$$M_{\max} = \frac{W}{4\pi}[(1+\nu)\ln\frac{R_S}{R'_L} + \frac{1+\nu}{2} + (1-\nu)(2R_S^2 - R_L'^2)/4R_O^2]$$

The maximum bending stresses, σ_{max}, is then

$$\sigma_{\max} = \frac{6M_{\max}}{t^2}$$

$$= \frac{3W}{2\pi t^2}\left\{(1+\nu)\ln\frac{R_S}{R'_L} + \frac{1+\nu}{2} + \frac{(1-\nu)(2R_S{}^2 - R_L'^2)}{4R_O{}^2}\right\}$$

Appendix 8

Fracture results of discs in 4-Ball test

Model No.	Specimen No.	Specimen dia. (mm)	Specimen thickness (mm)	Fracture load (N)	Fracture strength (Mpa)
1 pitch circle dia. = 30mm ball dia. = 10mm loading rate = 0.5mm/min	A 1	43.24	2.16	971	573
	A 2	43.24	2.18	935	543
	A 3	43.24	2.16	904	536
	A 4	43.24	2.18	930	541
	A 5	43.16	2.15	981	584
	A 6	43.32	2.16	935	553
	A 7	43.20	2.16	969	572
	A 8	43.22	2.20	1041	589
	A 9	43.24	2.20	1003	569
	A 10	43.24	2.19	976	560
2 pitch circle dia. = 30mm ball dia. = 10mm loading rate = 0.05mm/min	B 1	43.18	2.18	814	478
	B 2	43.20	2.18	909	529
	B 3	43.22	2.18	874	510
	B 4	43.16	2.18	902	526
	B 5	43.26	2.17	898	528
	B 6	43.24	2.16	848	506
	B 7	43.16	2.18	792	466
	B 8	43.26	2.16	895	531
	B 9	43.18	2.16	828	494
	B 10	43.24	2.16	888	528
3 pitch circle dia. = 30mm ball dia. = 10mm loading rate = 2mm/min	C 1	43.22	2.18	1047	603
	C 2	43.24	2.16	1004	591
	C 3	43.24	2.17	1078	626
	C 4	43.24	2.16	947	560
	C 5	43.28	2.15	1068	632
	C 6	43.24	2.15	1051	623
	C 7	43.16	2.17	1095	635
	C 8	43.40	2.13	993	602
	C 9	43.20	2.18	1078	620
	C 10	43.22	2.18	1096	630
4 pitch circle dia. = 40mm ball dia. = 10mm loading rate = 0.5mm/min	D 1	43.30	2.16	833	540
	D 2	43.30	2.15	822	538
	D 3	43.22	2.15	812	532
	D 4	43.34	2.15	892	581
	D 5	43.14	2.14	806	534
	D 6	43.28	2.20	970	600
	D 7	43.23	2.17	919	586
	D 8	43.36	2.19	808	510
	D 9	43.27	2.16	850	550
	D 10	43.22	2.16	891	575
5 pitch circle dia. = 40mm ball dia. = 5mm loading rate = 0.5mm/min	E 1	43.28	2.16	850	575
	E 2	43.20	2.16	817	555
	E 3	43.24	2.19	877	576
	E 4	43.26	2.18	879	583
	E 5	43.24	2.16	794	540
	E 6	43.23	2.17	917	612
	E 7	43.23	2.17	892	597
	E 8	43.17	2.16	832	564
	E 9	43.36	2.13	775	543
	E 10	43.38	2.18	838	557